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Mr. C. E. Capito has left for Trinidad.
Mr. W. H. Chalmers is on his way home from Burma.
Dr. E. Clark is home from Mexico.
Mr. L. R. Dalton is home from Peru.
Mr. T. R. H. Garrett has left for Java.
Mr. G. A. F. Grindle is home from Burma.
Mr. A. D. Jones has left Burma and is on his way home.
Mr. H. N. Sher has left Rumania and is in England.
Mr. H. N. Shorto has left California and returned to Mexico.
Mr. J. G. Smillie has left for Venezuela.
Mr. F. C. Waters is home from Persia.
Mr. W. Wallace Watt is home from Egypt.
Mr. L. J. Wilmoth has returned from Burma.

The Secretary will be glad to receive information as to the whereabouts of the following members:—C. A. Bauduin, E. Dawidson, J. C. Fortune, W. J. Harris, L. B. Holloway, W. Johnston, A. F. C. Parker, W. E. Sheperd and F. E. G. Watson.

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No.

Lubricants.*

By R. W. L. Clarke, B.Sc., F.I.C., F.C.S. (Associate Member).

The previous report in this series on the subject of lubricants dealt with progress up to about July, 1927. An endeavour has, therefore, been made in the present article to include the more significant information embodied in publications produced during the latter part of 1927, in 1928 and 1929. The chief advances made during this period would appear to be in the manufacturing and refining branches of the mineral oil industry. The problems of the cause of oiliness and the correlation of laboratory tests of a lubricant and its behaviour in service have received some attention, but only a little further illumination.

On the manufacturing side improvements continue to be effected in the distillation processes for the production of lubricating oils from crude petroleum. The chief aim of the modern process is to avoid as far as possible the protracted heating of a large mass of oil to high temperatures which is an inevitable condition of the old type of shell still operation. One of the first steps in this direction was taken some time ago by the Zieley[1] process in which the oil to be distilled is allowed to flow in thin films down inclined conduits situated in zones of successively increasing temperature. Vacuum is maintained in the conduits so that there is a constant disengagement of vapour. Cuts are made by separating the vapours from the conduits at different temperatures and by fractional condensation. The process does not seem to have been adopted to any extent, possibly owing to mechanical difficulties but more probably owing to the development of more efficient pipe still methods.

The pipe still type of plant, which was first employed in the petroleum industry as a topping unit, has now been developed for the production of lubricating oil cuts. The methods of operation are either of the single or double flash type. In the former case the pipe still and its accompanying fractionating equipment is fed with a whole crude, and gasoline, kerosine, gas oil and light lubricating distillates are taken off as overhead or distillate products in one operation. The character of the residue or bottoms is determined by the nature of the crude oil. This method of operation is being used for working up Pennsylvania oils to produce a steam refined cylinder stock as a residue product. In the case of the

* Paper received January 16, 1930.

[1] E.P. 248,513.

double flash method a reduced crude is prepared by taking off the lighter products in a pipe still plant operated at atmospheric pressures and at temperatures lower than those necessary in the first case. The reduced crude can also, of course, be produced in a conventional shell still topping bench. The reduced crude is now subjected to a second flash distillation and for this purpose it is heated to a high temperature, for example 700° to 800° F., in a pipe still and then injected into a combined vaporising and fractionating equipment. The vaporiser is operated at atmospheric pressure or under vacuum according to the volatility of the distillate cuts it is desired to produce. For the production of heavy distillates, such as cylinder stocks a vacuum of about 25 mm. may be maintained in the vaporiser as well as a high pipe still outlet temperature. The main advantages of this pipe still flash distillation are as follows: Owing to high velocities and turbulent flow in the pipe still, efficient heat-transfer obtains, the brief time of heating in which the oil is exposed to the maximum temperature for possibly not more than about one minute, minimises the destruction of valuable viscous products by cracking, also the vapours of the more volatile oils function in flash distillation in a similar manner to open steam in an ordinary distillation and have a carrying effect on those of the heavy oils.

Such plants are usually equipped with efficient fractionating towers of the bubble cap type which reduce entrainment of residue to a minimum and also act as deodorisers, particularly during vacuum operation. These factors tend towards the production of somewhat higher yields and better qualities of distillates. The following outline of one of the methods of operation with paraffin or mixed base crude oils is typical. The hot reduced crude from the pipe still is flashed into the evacuated vaporising section of the fractionating column. Gas oil passes off as a vapour from the top of the column. A light or medium lubricating oil cut, of suitable viscosity for dewaxing by cold pressing, is taken as a side-drawn stream from a plate near the top of the column. Cylinder stock is taken as a second side-drawn stream from an intermediate plate and tar or pitch bottoms are drawn from the base of the tower, usually after meeting a current of steam in a stripping section. The cylinder stock can be dewaxed by centrifugal methods. The pipe still method of distillation for the production of lubricating oils has been dealt with extensively by many authors among whom are Edmonds,[2] Bell,[3] Mulhaupt,[4] while four papers dealing with the practical and theoretical aspects of the subject were presented

[2] *J. Inst. Petr. Techn.*, 1928, **14**, 388.
[3] *Ibid.*, **14**, 88A.
[4] *Ibid.*, **14**, 171A.

at American Petroleum Institute meeting in December, 1928.[5] Two papers by E. Petty on "Wax-bearing Vacuum Distillates"[6] and "Vacuum Distillation of Lubricants"[7] describe the most recent American practice in the production of lubricants on these lines. The most recent developments in the Steinschneider high vacuum process for the distillation of lubricating oils embodies both pipe and shell still practice.[8] The procedure is as follows: The oil to be distilled is first heated to the desired temperature in a pipe still from which it passes to an evaporator tower or gas separator. The unevaporated portion is passed to a shell still fitted with a shallow tray extending practically over its entire length and width. The hot oil flows first on to the tray and then overflows into the still. The evaporating surface in the still is thus practically doubled. Heat is supplied to the shell still by the furnace gases from the pipe still. By maintaining a considerably higher pressure in the gas separator than in the still, light unsaturated odourous bodies are evaporated in the gas separator and lubricating distillates in the shell still under high vacuum. For the continuous treatment of large quantities a number of units can be operated in series.

Another method of avoiding the undue overheating of oils during distillation, which is described by Klemgard[9] and is also the subject of a patent by the Sun Oil Co.,[10] consists in heating the oil in a counterflow system by means of mercury vapour at a controlled temperature and pressure. The hot mercury vapours pass successively through a series of ten evaporators into the last of which hot crude oil, which has been topped in presence of caustic soda, is fed. The oil which is being fractionated and the mercury vapour pass in countercurrent directions through the respective chambers of the evaporators. The mercury vapour condenses as it gives up its heat to the oil and falls to the bottom of the mercury chamber from which it is re-circulated through the mercury heater, and thence to the first and hottest evaporator. It is stated that the oils produced require no further treatment. The latest development in the domain of distillation by indirect heating is the Govers process.[11] The indirect heating agent in this case is diphenyl vapour, which is circulated from a tubular boiler downwards through the space surrounding the tubes of the oil evaporator at a pressure of 110 lbs. per sq. in. and a temperature of 750° F. The diphenyl gives up its heat to the oil which is circulated upwards through the tubes

[5] *A.P.I. Bulletin*, X, (2), 33-66.
[6] *Refiner*, 8, (6), 62-66.
[7] *Oil and Gas J.*, 28, (25), 84, 111; *Refiner*, 8, (11), 90.
[8] E.P. 257,257.
[9] *J. Inst. Petr. Techn.*, 1928, 14, 158A.
[10] E.P. 305,846.
[11] *Refiner*, 8, (10), 63.

to an evaporating pan in which an absolute pressure of 5 mm. of mercury is maintained. Lubricating oil cuts are taken from the vapours leaving the pan. The condensed diphenyl is circulated from the base of the oil evaporator back to the diphenyl boiler. The complete Govers process which is in operation at an Illinois refinery consists in the vacuum distillation of topping plant residuum by the above method, followed by solvent extraction of paraffin, chemical treatment, filtration, and subsequently vacuum distillation for the finished lubricating oils in a similar indirectly heated distillation unit.

While dealing with the production of lubricating oils mention should be made of the Voltol[12], [13], [14] process. In this process a non-viscous oil is subjected to the action of high tension high frequency electric discharges while flowing over aluminium electrodes in a closed vessel at temperatures of 60°–80° C. The atmosphere in the vessel consists of hydrogen or nitrogen at an absolute pressure of 60–70 mm. of mercury. Electric current of a frequency of about 500 periods and at 4,500 to 12,000 volts pressure are supplied to the electrodes. The non-viscous oil, which may be of mineral, animal or vegetable origin, undergoes marked increases in viscosity after treatment for a number of hours. The magnitude of the viscosity increase is dependent on the time of treatment. Mineral oils also undergo a corresponding increase in flash point. It is stated that the oils thus produced possess good stability, low cold tests, low temperature viscosity coefficients, and are of much value for blending purposes with oils of lower viscosity.

The demand for oils of the best possible quality and of low cold test for aeroplane engine lubrication, and also as automobile oils for winter use, is directing attention to dewaxing processes. Oils of zero and lower cold tests have mainly been produced from naphthene-base crude oils, and it is now being more generally recognised that such products have the disadvantage of relatively high temperature-viscosity coefficients, and in some cases relatively high oxidisabilities. The more recent methods of producing zero cold test oils from paraffin-base crudes of the Pennsylvanian type consist in (*a*) chilling the oil-naphtha blend before dewaxing by Sharples centrifugal method to temperatures of about —40° F. instead of the more usual temperature of about —20° F.; (*b*) double centrifuging as advocated by Olsen ;[15] (*c*) low temperature filtration with the help of a filter aid, such as " Filter-cel " as in the so-called

[12] *Chem. Abs.*, **22**, 160.
[13] *Ibid.*, **22**, 3981.
[14] E.P. 305,553.
[15] U.S.P. 1,715,670.

Weir process which is described by Lederer and Beattie.[16] In the Weir process wax distillate or long residuum is diluted with naphtha in the proportion of 50/50 or 25/75 parts respectively, one half pound of filter-cel being added per gallon of stock. The mixture is cooled to about —40° F., and filtered either by means of a press or through a continuous filter by suction. The above authors state that the temperature differential between the filtration temperature and the pour point of the topped oil obtained from the filtrate is approximately 30° F. A considerable amount of experimental work is being carried out on the use of solvents other than naphtha for the dewaxing process. In this connection alcohols, ketones, benzol and chlorinated light petroleum products are being tried out. Mixtures of benzol and acetone or butanol seem to be working economically on a large scale in one or two installations.

A large number of papers have been published on methods of refining lubricating oils and reconditioning used oils. Abstracts of the most important of these have already appeared in this journal. The majority do not not call for special comment here as they embody minor modifications of accepted methods. The Edeleanu or sulphur dioxide process, which has found considerable application for the refining of kerosine is being applied to some extent for the refining of lubricating oils. In one patent [17] it is claimed that lubricating oils characterised by freedom from acids, reduced content of tar, good stability and resistance to oxidation are produced by liquid sulphur dioxide treatment followed by a distillation of the refined oil, or portion insoluble in the liquid sulphur dioxide, with one per cent. of sodium hydroxide. Liquid sulphur dioxide refining before the removal of paraffin by cold filtration is advocated in one patent 17[a] for the production of oils of low cold test. The use of an adsorbent clay, activated by acid treatment, in the form of a pulp or moist pasty mass appears according to a number of writers to be finding increasing application for the neutralisation and finishing of acid treated oils (see Kauffmann[18] and Reid[19]). The main features of the process were described at some length in the annual report on Lubricants in 1925.[20]

The problems of the pure chemistry of the mineral lubricating oils continue to receive some attention. Spilker[21] has carried out some synthetic research on the building up of highly viscous substances by the condensation of olefinic bodies and benzene homo-

[16] *Oil and Gas J.*, **28**, (29), 115.
[17] E.P. 307,649.
[17a] *J. Inst. Petr. Techn.*, 1929, **14**, 224A.
[18] *J. Inst. Petr. Techn.*, 1927, **13**, 239A.
[19] *Ibid.*, 1928, **14**, 20A.
[20] *Ibid.*, 1926, **12**, 403.
[21] *Petr. Zeit.*, **23**, 448.

logues. Pseudo-cumene-allyl alcohol was found to have a viscosity Engler at 50° C. of 16·2°. Viscosities of the hydrogenation products of polynuclear aromatic hydrocarbons have also been determined. The polyhydrides of chrysene have all the characteristics of lubricants, but are not identical with those present in petroleum oils. Bacon[22] has determined the bromine absorption of certain lubricating oils by the bromide-bromate method, and has calculated the proportion of unsaturated hydrocarbons from the results. Motor oils of Pennsylvanian origin were found to contain 3·7 to 5·7 per cent., while others from N. Texas and Salt Creek sources contain up to 8·2 per cent. of unsaturated bodies. On the other hand Mabery[23] finds that when petroleum hydrocarbons are treated with the calculated amount of bromine in carbon tetrachloride solution, one equivalent of bromine is absorbed and an equivalent quantity of hydrobromic acid liberated. It is therefore concluded that only substitution occurs, or, in other words, the oils do not exhibit evidence of unsaturation. This conclusion is difficult to reconcile with the accepted views on the composition of lubricating oils and also with the results of Johansen[24] and other investigators.

The problem of the composition of the lubricating oil constituents of petroleum has been further studied by Smith[25], who has worked on Salt Creek (Wyo) crude oil. Lubricating oil cuts are produced by more or less conventional laboratory methods, and separated by treatment with acetone into two portions, one relatively insoluble and the other extremely soluble in this solvent. The viscosity, refractive index, specific gravity, molecular weight and ultimate analysis of the fractions indicate the presence of two groups of oils. The group relatively insoluble in acetone has a low specific gravity-viscosity ratio, low carbon-hydrogen ratio and relatively low unsaturation. Members of this group are of the same type as Pennsylvanian light lubricants and their composition indicates that they belong to series having the general formulæ C_nH_{2n-2} or C_nH_{2n-4}. The other group, extremely soluble in acetone, has high viscosity-gravity ratios, high carbon-hydrogen ratios and considerable apparent unsaturation. Members of the latter group appear to belong to such series as C_nH_{2n-6} to C_nH_{2n-12}. Physical characteristics are found to vary uniformly with apparent changes in constitution, such as increased unsaturation. The author in his concluding remarks compares the results now obtained with those of other investigators. In this connection it will be remembered that

[22] *Ind. Eng. Chem.*, **20**, 970.
[23] *J. Amer. Chem. Soc.*, **48**, 2663; **49**, 1116.
[24] *Ind. Eng. Chem.*, 1922, **14**, 288.
[25] *U.S. Bur. of Mines Tech. Paper*, No. 428 (1928).

Mabery[26] separated Sour Lake, Cabin Creek, Mecca and other crudes into two groups of fractions by alcohol-ether extractions and Alibone and Wilson[27] carried out similar work on Burmah and Hardstoft crudes by extraction with a mixture of acetone and liquid sulphur dioxide. In both cases a separation into more soluble unsaturated hydrocarbons and less soluble more saturated hydrocarbons was effected. Mabery's residual hydrocarbons obtained from Cabin Creek lubricating fractions show much less divergence from the soluble fractions than do the similar fractions now obtained from Salt Creek oils by the acetone method.

A certain amount of work has been carried out on the physical properties of lubricating oils which should be noted. Winchester and Reber[28] have determined the density and surface tension of some oils at a number of temperatures up to about 300° C. The curves connecting density and temperature, and surface tension and temperature are straight lines which are substantially, but not quite, parallel for oils from different sources. Rowell and Findlayson[29] have measured the viscosities of a number of lubricating oils at temperatures ranging from 10° to 250° C. The results of their measurements are given as absolute viscosities and as fluidities. It is shown that within certain limits the square root of the fluidity of an oil is a linear function of the temperature, while at lower fluidities a linear relation exists between the cube root of the fluidity and the temperature. Attention is drawn to the necessity of subjecting the oil to a preliminary heat treatment before concordant values for the viscosity of an oil at high temperatures can be obtained. It is not clear whether the change in viscosity during the prolonged heating, which was carried out in the capillary viscometer itself, is a physical or chemical effect. Walther[30] obtains results for American and Russian oils which indicate that the logarithm of the logarithm of the viscosity of an oil in degrees Engler is a linear function of temperature. Ostwald and Föhre[31] have made a study of the pressure-viscosity of 17 different oils, and also of mixtures of some of the same oils with oleic and acetic acids. The observations were made at 25° C. in a capillary viscometer under pressure heads of about 5 to 100 cms. of water. All the straight oils except two more viscous "Voltols" conform to the Hagen-Poiseuille law, giving substantially constant values for the product pressure x time for the out-flow of an equal volume. The two "Voltols" exhibit

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[26] *Ind. Eng. Chem.*, **15**, 1233; **16**, 207.
[27] *J. Inst. Petr. Techn.*, 1925, **11**, 177.
[28] *Ind. Eng. Chem.*, **21**, 1093.
[29] *J. Inst. Petr. Techn.*, 1928, **14**, 402.
[30] *Ibid.*, 1928, **14**, 330A, 331A.
[31] *Ibid.*, 1929, **15**, 69A, 71A.

a decrease of apparent viscosity with rising pressure. This is considered to be due to "structural" viscosity such as that shown by colloidal solutions. The ordinary lubricating oils do not exhibit evidence of colloidal nature by this method of investigation. The addition of oleic and acetic acid to an oil caused a marked reduction in the pressure viscosity. It is well known that mechanical treatment alters the viscosity of certain colloidal solutions, so the effect of mechanical rubbing or grinding on the viscosity of four of the oils was also investigated. The mechanical treatment caused a rise in viscosity of about 3 per cent. in the case of two machine oils, and about 29 per cent. in the case of an automobile oil. It is interesting to note that there is an increasing tendency to regard the viscosity-temperature coefficients of lubricating oils of major importance. Robinson and Scheumann[32] give the values of this coefficient in terms of the slope of the log. viscosity vs. log. temperature curve for oils of Pennsylvania, Mid-Continent and South Texas origin. The values thus calculated vary from – 2·04 for Pennsylvania oils to—3·30 for S. Texas oils. The term viscosity-index is coming into vogue for expressing this characteristic of oils. Dean and Davis[33] state that the viscosity-index is independent of the actual viscosity, and is a constitutive property of the oil. The viscosity indexes of all lubricating fractions from any given crude are ordinarily approximately constant. These authors have developed a method of calculating the viscosity-index which is simple, but requires the use of a special table of values. Hatschek has published a comprehensive treatise on the viscosity of liquids (See review *J. Inst. Petr. Techn.*, **15**, 135).

The carbonisation of oils suitable for cylinder lubrication, particularly of internal combustion engines, has been the subject of a considerable amount of work. Donaldson[34] has approached the question from the point of view of vaporisation and has shown that the amount of evaporation occurring at a given temperature is equally dependent on the area exposed and on the time of exposure. It is concluded that it is advisable to use as light-bodied an oil as the lubrication conditions will permit so as to obtain the smallest quantity of carbon deposits. Livingstone, Marley and Gruse[35] have continued their investigations on the carbon deposits in internal combustion engines and have compared the carbon forming tendencies of four oils of similar viscosities, two of which are distillates and two distillate-bright stock blends. The results show that under similar conditions the blended oils

[32] *Oil and Gas J.*, **28**, (17), 40.
[33] *Chem. Met. Eng.*, **36**, 618.
[34] *J.S.C.I.*, **46**, 324T.
[35] *Ind. Eng. Chem.*, **18**, 502.

caused the formation of about one and one-half times the amount of carbon as did the distillate oils. In a summary of their work[36] on this question with seventeen oils, these authors conclude that the Conradson carbon residue test is a fairly reliable indication of the carbon-forming tendency of the oils in a poppet valve engine. The agreement is more marked with paraffin-base oils. A lengthy series of observation on road vehicles equipped with sleeve-valve engines lubricated with (*a*) a high carbon paraffin-base oil, (*b*) a low carbon paraffin-base oil, and (*c*) a naphthene-base oil showed that a paraffin-base oil of very low carbon residue is only slightly better than one of normal carbon residue and both are strikingly inferior to the naphthene-base oil. The reason for the marked difference is ascribed to the fact that the carbon from the naphthenic oil is of soft nature and easily eroded away by the exhaust gases from the ports of the sleeve-valve engine.

Kelly[37] has compiled a useful and interesting survey of the views and results of many workers in the field of lubrication particularly with reference to the petrol engine. The same author[38] has published his experience on the Ramsbottom Method for the determination of the carbon residue value of oils, which is considered to be superior to the Conradson Method. Suitable apparatus and the method of operation of the test are dealt with very completely.

The influence of the viscosity and the cold test (solidifying point) on the lubrication of cold internal combustion engines has been the subject-matter of a number of publications. Some writers have called attention to the fact that oils can be circulated by an immersed pump or drawn up by a suction tube at temperatures of 10° to 15° F. below their pour points. A general paper, on engine lubrication by Pomeroy[39] is of interest; in it he deals with the increase in efficiency of a petrol engine, as measured by a dynamometer, which occurs with rise of the water-jacket and lubricating oil temperatures. Barnard[40] describes results which show that the cranking effort necessary to rotate a cold engine depends almost entirely on the viscosity of the oil apart from its cold test. In the course of the investigation the viscosity of the oils used was determined down to temperatures of 0° F. under different pressure heads. None of the oils examined followed the laws of viscous flow at low temperatures. The cranking effort

[36] *Ibid.*, **21**, 904; see also *J.S.A.E.*, **13**, 607; **20**, 688.
[37] *J. Inst. Petr. Techn.*, 1928, **14**, 115.
[38] *Ibid.*, 1929, **15**, 495.
[39] *Ibid.*, 1929, **15**, 73A.
[40] *Ind. Eng. Chem.*, **20**, 843.

appears to depend on the viscosity of the oil corresponding to fairly high shearing stresses (say 20 to 30 kilos. per sq. cm.).

The formation of acids in crank-case oils during service has been studied by Meston[41]. Tests show that the acidity increases steadily to a definite degree, after which an apparently stable state is reached. Dilution also increases rapidly to a steady value which depends on mean temperature and humidity. Haslam and Frölich[42] have investigated the deterioration of mineral oils using a highly refined P.L. alone, in presence of positive catalysts such as copper oxide and in presence of negative catalysts such as diphenyl hydrazine. Ultimate analyses of the original and oxidised oils showed that the latter contain 11 to 12 per cent. of total oxygen of which about 2 to 3.5 per cent. is present as part of acidic constituents. Pidgeon and Tester[43] have determined the characteristics and percentage dilution of samples of the same oil after use in a number of automobile engines. The figures for percentage dilution range from 2 to 14 per cent. Curves showing the effect of dilution on viscosity, specific gravity and flash point are given.

A number of papers have appeared on the subject of the stability of transformer and turbine oils. Quite a number of these consist of a general survey of the subject without any new experimental evidence. Brian Mead and his collaborators[44] have determined the deterioration of such oils by static methods at temperatures of 120°, 140° and 150° C. Factors such as the surface area exposed, amount of oxygen absorbed and acidity developed are dealt with as well as sludge formation. The effects of 177 different substances were tried as catalysts for sludge formation. Some 48 substances were found to have inhibitory effect, but no general conclusion as to the types of compounds which act as anti-oxidants could be drawn.

Ford[45] has endeavoured to correlate the oxidisability of transformer oils with the proportion of unsaturated bodies present as determined by the amount absorbed by treatment with 150 per cent. of concentrated sulphuric acid. The tendency to sludge increases with increased sulphuric acid absorption. Ford also suggests that the naphthene content as determined by the aniline point is of importance, while Heyd[46] advocates the use of refractometric and absorption spectra measurements in estimating the qualities of insulating and lubricating oils.

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[41] *Ibid.*, **19**, 312.
[42] *Ibid.*, **19**, 292.
[43] *J. Inst. Petr. Techn.*, 1929, **15**, 91.
[44] *Ibid.*, 1927, **13**, 303A.
[45] *Ibid.*, 1927, **13**, 307A.
[46] *Ibid.*, 1927, **13**, 244A.

In connection with the theoretical aspect of lubrication, the publication of the work *Contribution à L'Étude du Graissage* by P. Woog is of much interest[47].

The subject of the manufacture and properties of lubricating greases has received some attention. Klemgard[48] describes the advantages possessed by a pressure method of preparing the soap base over the open kettle method. The same author[49] has published a comprehensive monograph dealing with the manufacture and properties of lubricating greases. Further information concerning modern methods of grease manufacture and testing will be found in the abstracts of four papers given at a Symposium on Grease[50].

The characteristics and manufacture of cutting oils are discussed by Cupit[51] and Hudson[52] deals with the difference in efficiency between soluble and straight cutting oils and points out the advantages of the latter for taking heavy cuts. Hart[53] gives the results of investigations on water miscible mineral oil preparations, in which sodium oleate and Turkey red oil are used as emulsifiers. The necessity of the presence of a critical amount of free oleic acid and methods for determining the relation between the amounts of oleic acid and oil as well as suitable tests for the stability of the products are discussed.

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[47] *Ibid.*, 1927, **13**, 241A.
[48] *Ibid.*, 1929, **15**, 132A.
[49] *Ibid.*, 1928, **14**, 150 (review).
[50] *Ibid.*, 1929, **15**, 352A, 354A, 364A.
[51] *Ibid.*, 1928, **14**, 324A.
[52] *Ibid.*, 1929, **15**, 68A.
[53] *Ibid.*, 1929, **15**, 67A.

Heavy Oils and Lubricants.*

By William Lee (Member).

No new process appears to have been introduced during the last two years for either the production of heavy distillates or their treatment to make them available as lubricants. Notwithstanding a considerable amount of work on lubricating distillates the present methods of obtaining and treating them have all been in existence for some time, and improvements have been confined to extending their use and increasing their efficiency.

The extension of distillation under high vacua[1], whereby a greater yield of better quality heavy distillate is obtained, has resulted in the production of excellent lubricants of high viscosity which previously were scarcely suspected in the crude and were lost in the large quantity of cracked products produced when attempts were made to carry atmospheric pressure distillation with superheated steam to extremes. Uncracked, clean and useful products are available in larger quantity. Naphthene base oils have up to the present yielded the best results. In this field Russian producers seem to have lagged behind and experiments are as yet in the laboratory stage[2]. The present heavy Russian lubricating oil seems to be confined to "O.O." Russian, which shows evidence of cracking and is not what it should be. Under vacuum distillation this oil should be obtained free from tar spray and decomposition products, and the less waxy varieties of Russian crude should yield more viscous fractions carrying with them their characteristic greater stability than similar distillates from heavy gravity American crudes.

By washing certain of the crudes with alkali before distillation, distillates are obtained which require only filtration, and we have now become familiar with Texan type oils of very pale colour having considerable stability under moderate heat and air conditions, excellent demulsibility, freedom from acidity, and better flash-point and cold test than the earlier oils.

The old acid and alkali treatment of lubricants has been increasingly displaced by the "Contact Filtration" process[3] and by the extension of the Edeleanu liquid sulphur dioxide process[4] to lubricants. The name "contact process" was at first applied (1868) to the method of adding selectively absorptive earth in finely divided form to the oil to be refined, as distinct from the more

* Paper received January 3rd, 1930.

common method of running distillates through filtering columns of granular earth. One advantage is obvious, namely, the production of a uniform material. A deleterious substance can be removed uniformly from the whole bulk of the oil by contact filtration, whereas the tower filtration method unnecessarily purifies the first runnings, while the last may be little altered from the original oil.

A more effective modification of the " contact process " involves the use of specially treated and activated earths. These are prepared by the action of sulphuric acid on the earths followed by a thorough washing with water, drying and grinding. In this form the earths can be used to avoid the alkali treatment after the acid treatment, since it is capable of absorbing any unsettled acid tar and the acid products. The oils first marketed from this process were characterised by considerable irregularity in colour, odour and acidity, due to the difficulty experienced in the precise control of the acid attack, the settling of the tar, and in determining the correct amount of filtering medium and the optimum temperature. Unless the whole of the products of the acid treatment are thoroughly absorbed the refining action is largely defeated by the decomposition products formed on raising the temperature prior to filtration. The difficulty appears to have been overcome and uniform neutral grades of good colour are now available. The unpleasant phenomenon of separation of both crystalline and gelatinous sulpho-compounds from high gravity lubricants on storage has almost disappeared in the last two years.

The use of the Edeleanu liquid sulphur dioxide process applied to the production of lubricants is on the increase. The author is indebted to Dr. Ion Edeleanu for the following :—

The year 1929 marks a further progress in the utilization of the Edeleanu Process. The Nederlandsche Koloniale Petroleum Maatschappij has substantially extended its existing plant in the Dutch Indies. The first extension of this plant to the extent of about 300 tons has already been put into operation. The further extension is now under erection and will be put in operation in the spring of 1930. New plants were erected during the year by the Bataafsche Petroleum Maatschappij in Pladjoe and at Pangkalan Brandan. Two smaller plants were put in operation in Trinidad. In the United States of America three big plants were put up and their operation started both in California and in Texas. These three plants have a daily capacity of about 1500 tons. A further plant for lubricating oil will be put in operation in December, 1929. This unit is provided for the treating of 350 tons of lubricating oil per day. It is noteworthy indeed that the oil industry is devoting increasing attention to the treating of lubricating oil with the Edeleanu process.

In addition to the plants which have been put in operation during the year under review there are a number of other big plants under construction. Among others there is in construction a second plant of a daily capacity of 1000 tons for the Anglo-Persian Oil Company for Persia, another plant for Great Britain, then a plant for Mexico and a further very big plant with a daily capacity of some 1000 tons to be erected on the Californian coast.

A fairly complete account of the plant and process is given by R. Brandt in the *Oil and Gas J.* for March 7th, 1929.

Sulphur dioxide most readily removes the aromatic hydrocarbons, olefines, cracked oils, sulphurised and oxidised compounds, and the oils at present marketed have been treated by a suitable choice of temperature and quantity of solvent to remove these bodies.

By modifying the method of working a much more fundamental separation of the same oils is possible and heavy naphthenic oils yield lighter fractions approximating to Russian oil in characteristics. The improvement in the Mexican oil, mentioned below in connection with turbine oils, is likewise most marked, as also is that in the Columbian examples in Table I.

The general claims for the process are that in comparison with the oils obtained by the older sulphuric acid and soda treatment the new oils are lower in specific gravity, sulphur content, carbon value, and have improved resistance to oxidation, flatter viscosity-temperature curves, better demulsification, and the oils retain their colour for a longer time. It is to be noted that these claims are comparative and point out the improvement in oils obtained by the one process over those obtained by the other. Other crudes may yield oils which are better without the application of this process.

Naphthene base oils have been very much improved and larger quantities are put forward as being suitable for turbine lubrication, for which purpose the Russian especially are valuable, although their life under difficult conditions is not so long as that of the paraffin type.

Attempts are being made to make refining processes continuous. This is readily accomplished with solvent processes, but is more difficult with methods of chemical treatment. Progress is however, being made in continuous acid treatment, removal of tar by centrifuging, addition of activated earth and subsequent filtration.

The above methods have been applied more especially to the treatment of asphalt-base oils, but it is claimed that both processes are applicable to oils of paraffin base[5]; with Pennsylvanian type increased activity has been shown in the application of chilling and centrifuging processes[6] and larger quantities of oils of paraffin type, of high viscosity, lower cold test, much lower coke and greater stability to oxidation have been put forward. The distinction between heavy distilled oils and so-called residual oils is being gradually narrowed. As an illustration of what has been done the following oils are worth examining. They are all of pale yellow colour and quite clear at ordinary temperatures.

TABLE I.

COLUMBIA DISTILLATES BEFORE AND AFTER REFINING WI

(*a*) *Before*; (*b*) *after treatment.*

	Turbine Dist.		Medium Machine.		Machine.	
	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	
Yield, per cent. ..		81·5		81		7
Colour N.P.A. ..	Opaque.	No. 2.	Red/bn.	No. 3.	Opaque.	N
Spec. Gravity 15° C.	0·918	0·894	0·925	0·901	0·928	
Viscosity Engler—						
At 20° C.	34·7	24·0	—	—	—	
,, 50° C.	5·3	4·45	10·4	8·0	13·0	
,, 100° C.	—	—	1·9	1·8	2·05	
Open F. Point ..	210	210	229	228	236	2
Set Point below ..	—20	—20	—20	—20	—20	—
Acidity mg./KOH/gm.	2·6	0	2·2	0	2·2	
Sulphur Content ..	0·91%	0·41%	0·87%	0·54%	0·87%	
Ash	—	—	0	0	0	
Asphalt	—	—	0	0	0	
Tar Value	—	—	14%	5%	20%	
Conradson Coke ..	—	—	0·3	0·1	0·31	
Oxidation Value—						
50h, 120°, no 0_2 ..	—	0·12	—	—	—	
70h, 120°, 0_2 ..	—	0·13	—	—	—	

Tar Content.—50 c.c. oil dissolved in 50 c.c. normal benzine, shaken for 5 mins. wit
the percentage of oil dissolved in the acid is measured.

Open Flash Pt.—Marcusson's Cup.

Colour.—National Petroleum Association's numbers.

TABLE I.

KA DISTILLATES BEFORE AND AFTER REFINING

(*a*) *Before*; (*b*) *after treatment.*

ne Dist.		Medium Machine.		Machi	
	(*b*)	(*a*)	(*b*)	(*a*)	
81·5			81		
No. 2.		Red/bn.	No. 3.	Opaque.	
0·894		0·925	0·901	0·928	
24·0		—	—	—	
4·45		10·4	8·0	13·0	
—		1·9	1·8	2·05	
210		229	228	236	
—20		—20	—20	—20	—
0		2·2	0	2·2	
0·41%		0·87%	0·54%	0·87%	
—		0	0	0	
—		0	0	0	
—		14%	5%	20%	
—		0·3	0·1	0·31	
0·12		—	—	—	
0·13		—	—	—	

ed in 50 c.c. normal benzine, shaken for 5 mins. wi
e acid is measured.
up.
ssociation's numbers.

LE I.

ER REFINING WITH EDELEANU PROCESS.

after treatment.

	Machine.		Cylinder Oil.		Transformer Oil.	
	(*a*)	(*b*)	(*a*)	(*b*)	(*a*)	(*b*)
		78		83		79·7
	Opaque.	No. 4.	Dk. gn.	No. 6.	Dk.	No. 1.
	0·928	0·902	0·939	0·912	0·895	0·873
	—	—	—	—	4·3	4·04
	13·0	9·7	45·3	25·4	1·7	1·7
	2·05	1·9	3·7	3·0	—	—
	236	238	271	275	152	155
	—20	—20			—20	—20
			—15	—7		
	2·2	0	1·0	1·0	4·4	0
	0·87%	0·48%	1·22%	0·59%	0·58%	0·28%
	0	0	0	0	—	—
	0	0	0·13	0	—	—
	20%	8%	25%	13%		
	0·31	0·14	1·86	0·59		
	—	—	—	—	—	—
	—	—	—	—	—	0·03

for 5 mins. with 10 c.c. H_2SO_4. After standing several hours in W.B.

TABLE II.

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
Sp. Gr. at 15° C.	0·885	0·8865	0·889	0·891	0·893
Closed Flash-Point—°F. ..	330	440	480	530	555
Viscosity (Redwood. secs.) at					
100° F.	695	1160	1640	2200	3280
140° F.	213	342	466	618	870
200° F.	73	100	131	157	206
Cold Test—°F.	5/10	0/5	5/10	10/15	below 20° F.
Coke Value at 550° C. ..	0·357	0·242	0·307	0·368	0·480
Blowing Factor 12 hrs. ..	1·721	1·722	1·461	1·436	1·337
Sulphur %	0·18	0·18	0·19	0·20	—
Acidity—mg/KOH/grm. ..	0·093	0·123	0·105	0·056	0·041
Saponification Value— mg/KOH/gm. ..	0·45	0·72	0·34	0·1	2·07
Demulsibility I.P.T.—mins.	8	10	7	6	drops out immed.

The utility of oils of the above type for use in internal combustion engines is at once evident. The coke value is as low as oils of equal viscosity at 200° F. of the heavy distilled type. The temperature-viscosity gradient is better, the cold test is as good or better, and the fear that appertains to distilled oils, namely, that at high cylinder temperatures the oil may either evaporate too readily or be consumed too rapidly, is very considerably lessened.

Production of Heavy Lubricants by Polymerization.

During the current year there has been a revival of the electrical process patented by the Count de Hemptinne. This process consists in exposing mineral or fatty oil in thin films to an alternating high voltage discharge through an inert gas at low pressure, and would appear to depend on the bombardment of the oil molecules by the cathode ray. Saponifiable oils, particularly rape and whale oils, are rapidly built up into compounds of huge molecular weight of the order of 6000. Mineral oils are polymerized with more difficulty, but useful oils of commercial interest can be produced from both Pennsylvanian and Western Bright Stocks, and claims are made that the blends of the condensed products have a much greater "oiliness" than that of the natural mineral oils. Friction experiments at high pressure and slow speed certainly seem to bear this out, and the claim may be scientifically sound as it appears highly probable that one factor in "oiliness" is high molecular weight and molecular volume.[7]

These condensed oils are marketed in Germany under the name of "Voltol," and in Belgium and certain other countries under the name of the "Elektrion" oils. The earlier products undoubtedly suffered from instability at high temperatures; mixtures of

elektrionized rape and a thin mineral oil if maintained at comparatively low temperature of 212° F. steadily lost a serious amount of viscosity. In the later products this appears to have been overcome, as similar exposure of the blends made with recent oils showed no diminution.

The claims made are distinctly interesting and worth investigation.

An account of the process and its products is given by K. Wolfe in *Petr. Zeitung*, 1929, **25**, 95.

Recovery of Used Oils.

Remarkable activity has been shown during the past few years in patenting processes calculated to recover and render usable oils which have done service principally in turbines and internal combustion engines.

The subject divides itself naturally into two parts, methods involving an attempt to purify the oil during use, and those necessitating the removal of the oil and purification in bulk elsewhere before further use.

In the first category, constituting the most practical and really effective means, is the use of a centrifuge, whereby the whole or part of the oil in circulation is centrifuged clear of water, foreign impurities, carbon and asphalt of dimensions which matter, leaving a small amount of impalpable asphalt and carbon which does not seriously affect either the lubricating value of the oil or the amount of carbon produced on coking. This process is no longer effective when the oil becomes saturated with soluble oxidation products of gummy resinous and acidic nature. When oil is circulated or repeatedly used and liable to take up water, dirt, or become oxidized as in turbines, internal combustion engines, etc., some form of centrifuge should be fitted. The application of this process is now imminent on road vehicles. The use of filters, with or without improvised distillation apparatus[8], cannot possibly yield satisfactory results. Filters become choked with wet sludge; the distillation is not controlled and is ineffective in the presence of much water; the further exposure to heat only causes a more rapid deterioration of the oil.

When the oil has reached the condition in which further centrifuging makes little improvement it requires thorough refining. Precipitations with sodium silicate, glue, soap, Fuller's earth, caustic soda, salt, oleic acid, oxalic acid as proposed in many patents[9] are clumsy, wasteful and amateur in character, and none of them deals adequately with the soluble oxidation products.

Application of existing refinery methods[10] returns the oil almost to its original condition. Thus by centrifuging out heavy solid matter, drying, distilling off light oils and treating the remainder

with carefully regulated amounts of sulphuric acid followed by filtration, perfectly wholesome oils are obtained free from oxidation products. Treatment with anhydrous aluminium chloride is very effective. A slight excess of the chloride is added to the dry waste oil to attack the soluble and insoluble impurities. A tar forms on heating and leaves the oil in a paler and well refined condition. Heating is continued to agglomerate the tar which decomposes, yielding petrol and light oil and a deposit consisting of carbon and alumina. The oil easily filters free from solids but may need slight topping to remove non-lubricating hydrocarbons.

When crank case oils have been contaminated with greases they are difficult to purify, and the recovered oil has a low commercial value, being only fit for fuel or common grease-making. Oils which have not had a prolonged stay in a crank case are rendered fit for use by clay filtration only. Oils which have been in use a long time are usually unfilterable as they clog the filter when pressure is applied.

The following illustrates the results of various treatments on a large scale of a crank case oil, using the coke value as an index of the degree of recovery.

Treatment.	Coke value at 550° C. %
Blackened oil from crank case ..	2·3
Filtered only	0·77
Filtered and bleached	0·65
Acid treated and filtered	0·52
Original oil	0·45

The treatment on a small scale with aluminium chloride gave a perfectly clean red oil of better colour than the unused oil, and the coke value was only 0·3 per cent. From the yield of light oil and petrol (amounting to 10 per cent.) obtained on distillation it was evident that the amount of treatment could have been cut down.

Correlation of Laboratory Tests with Results Obtained in Practice.

Work has been continually going on in the above direction, but on the whole no great advance has been made. Directions in which to conduct research are becoming more clearly defined, but in none can it be definitely said that absolutely reliable laboratory tests have been evolved.

Oxidation.

The subject which has received most attention is naturally concerned with oxidation, as the life of a lubricant depends very largely upon its resistance to the action of atmospheric oxygen at various temperatures, according to the machine in which the oil is working.

That oxidation plays an important and destructive rôle in the crank-cases of engines is shown by the work of King and Mardles,[11] and others who have patented the addition of certain anti-oxidation catalysts to the lubricating oil. They show the same oil run with and without the anti-catalyst under exactly similar conditions. The untreated oil gives the usual black opaque sludge whereas the sample from the crank-case using the treated oil is only slightly darkened and is still transparent and red. The same catalyst seems to have a beneficial effect on the nature of the coke, as after using the treated oil the pistons are almost clear of carbon; either the anti-catalyst causes a difference in the line of cleavage during destructive distillation, or the absence of oxygen from the decomposing oil results in less carbon. This strikes a very important note and more should come of it. It would appear that the material used should be more or less volatile so that the oxidation of the oil fog may be prevented.

Seyer and Allen[12] have attempted to trace the action of oxygen on crank case oil by taking the iodine value of used oil and comparing it with that of unused oil. They have tested about sixty samples and find that with few exceptions (due to crank-case dilution) the iodine values decreased. The average decrease after 1,000 miles is about 2 in the iodine number. The iodine numbers of the oils vary from 14·9 to 47·3, but very few oils of low iodine value were used. Both high and low iodine oils gave high or low reductions in value so that no correlation was apparent.

In the turbine, oil becomes useless and sometimes corrosive as the amounts of oxygenated products, asphalt, acidic and aldehydic substances increase. In the crank-cases of internal combustion engines such as the Diesel and the petrol engine, it is the action of atmospheric oxygen which brings about the formation of the black precipitate usually designated carbon. This carbon is distinct from the destructive distillation carbon (Conradson Coke) as it is plain that the temperature in the crank case is much below coking temperature, while analyses show that it contains oxygen and hydrogen as well as sulphur.

In the average I.C. engine crank-case oxidation is going on at two distinct rates; first a small quantity in the form of mist or fog is severely oxidised, producing insoluble asphalt which is transformed later into "carbon"; and second, slower and milder oxidation of the bulk of the oil in circulation at a lower temperature. The tendency to form coke on the piston tops, cylinder heads, behind rings, is not a simple function of the Conradson type of carbon, but is modified according to the degree to which the crank case oil has been oxidised before being forced up into the cylinder. If the oxidising action is slight or if the consumption is heavy, the

oil is not much changed, and up to a point, the Conradson carbon is a rough indication as to what can be expected above the piston. This amount is also affected by the type of hydrocarbon and the degree of refinement. Moderately refined oils of Pennsylvanian type give a hard adherent coke; naphthenic oils, particularly those of low specific gravity give a non-adherent carbon, which breaks off and is blown out of the exhaust before it has reached any serious thickness; well refined Pennsylvanian oils are intermediate, and can be obtained with very low carbon content, but still such carbon as they give tends to the hard side. This can be modified, and a mixture of naphthenic and low carbon paraffin is indistinguishable in behaviour from viscous naphthenic oils alone.

It is obvious that the appearance, behaviour and condition of a petrol engine is far from being quantitatively related to the coke value of the original oil. Many oils of very low coke value give an extremely dirty crank-case, although above the piston the condition may be fairly good. The only factor appertaining to the oil which could account for this is proneness to oxidation, and it is generally admitted that some oxidation test is necessary for the evaluation of an oil, but there is no general agreement up to the present as to what that oxidation test should be.

It is easier to state what types of oxidation tests are bad. Heating in vessels in an air oven is about the worst type, as the distribution of temperature, even with revolving shelves, is not uniform. The amount of air going through the oven is usually unmeasured and not easy to measure. The number of samples present at a time is not invariable, and different types are often attempted at the same time. The nature of the oxidising atmosphere is therefore variable, and may contain varying amounts of oxygen in addition to oil vapour, some untouched and some in various conditions of oxidation.

The surface exposed in relation to the volume of oil is not always specified, and the nature of the vessel containing them is seldom fixed. It would appear that no method is satisfactory unless each sample is done individually in its own vessel, with fixed conditions as to the quantity of oil, area of exposure, amount of air passed and close regulation of the temperature.

It is a distinct advantage to have the current of air rapid enough to agitate thoroughly the oil and so minimise the importance of the reaction which takes place between the top surface of the oil and the ambient atmosphere.

The nearest approach to these conditions is met by the English Air Board Test. This answers satisfactorily in the case of high boiling point oils which evaporate comparatively little and approximately equally, but is liable to serious error when more volatile

oils are tested. The effect of the loss of oil by evaporation is easily much more than the effect of oxidation on the viscosity test. The percentage of oil lost frequently exceeds 6 and sometimes over 12 on even rather heavy oils. The effect on the coke value is not so serious, being confined to an increase equal to the percentage loss in oil evaporated whereas the viscosity factor may be doubled.

The temperature of 200° C. is one at which all oils are extremely sensitive to small variations. The temperature of 150° C. is less critical and gives useful results with even six hours blowing. The test can also be combined with a metallic catalyst test. A definite area of copper or iron present during the blowing test, gave in the case of iron double the viscosity of the unblown oil, and in the case of copper an increase of five times. This suggests a useful line of research.

The following is a comparison between results obtained by such an oxidation test and those obtained in practice where the oil was used on ring-oiled bearings of electric motors.

TABLE III.

	No. 1 Oil.	No. 2 Oil.	No. 3 Oil.
Viscosity (Redwood, secs.) at 140° F.	133	133	135
Acid Value before use.	0·01	0·02	0·17
Acid Value after use	0·05	0·094	0·28
Asphalt after use ..	Nil.	Trace.	0·2
Blowing Factor at 150° C.	1·37	2·6	4·0
Result in bearing ..	After 4 months still clear and bright.	After 4 months cloudy and dark coloured.	Bearing ran hot after 3 months. Oil cloudy and black.

Butkov (*Erdöl und Teer* 1927, **3**, 123) has used a method of oxidation whereby a quantity of oil is exposed in a bomb to 15 atmospheres pressure of oxygen and maintained at a temperature of 150° C. for 6 hr., and Chernojookov (*Industrial and Engineering Chemistry* 1929, **21**, No. 4), has made use of the method to evaluate the suitability of oils for internal combustion engines.

The results given appear to be consistent, but the present writer happens to have been experimenting about two years with the same method and had arrived at the same temperature, pressure and time of heating as being most suitable, but the results obtained were not as regular as those obtained in the Mitchie oxidation test. In some cases with transformer oils, using oxygen, the contents fired, so recourse was made to air only; again the irregularities were difficult to eliminate. The principal difficulty lies in the effect of the lining of the bomb. The most promising apparatus up to the present is one of stainless steel into which a movable silica lining can be passed.

Livingstone and Gruse (*Journal of Industrial and Engineering Chemistry*, 1929, **21**, 904) give a continuation of their work of correlation between laboratory carbon tests and resulting engine conditions. They select three oils of approximately equal viscosity, namely 105-secs. Saybolt at 210° F., of three types, namely (1) high carbon paraffin, (2) low carbon paraffin, (3) naphthenic. The Conradson carbon contents of these were (1) 1 per cent., (2) 0·15 per cent., (3) 0·7 per cent.

Each oil was run on similar work on chars-a-banc with sleeve valve engines; six vehicles were run approximately each 10,000 miles, on each of the oils. The criterion used as to utility was the number of miles a vehicle would run before the power fell off owing to the valve ports becoming choked with carbon deposit which had built itself up across them. The figures obtained were (1) high carbon paraffin—314 miles; (2) low carbon paraffin—401 miles; (3) naphthenic—1556 miles; thus showing very little correlation between the coke content and the length of service.

The test, if it shows anything at all, indicates merely the degree of adherence of the cokes produced from the respective oils. No mention is made of the coke on the cylinders or pistons, or effect on rings or condition of crank-case. The same oils run on a laboratory poppet valve engine give results similar to the work mentioned in the previous papers, namely, that the cokes obtained on the piston stand roughly in the same order as the coke values of the oil used but are in no way proportional. The figures obtained in this case are (1) 8·6, (2) 5·9, (3) 5·3.

Only about 250 c.c. of each oil was used. A test of the latter kind is naturally a crude coke-content test. With the same engine, oils of the same viscosity, with the same oil-consumption and an engine run under the same conditions over a short time, one can expect approximately the same amount of oil to get to the top of the piston to be coked, and, provided the peeling stage has not been reached, one might expect carbon amounts in the same order as the coke values. If peeling of the carbon has occurred, any kind of

results may be obtained according to whether desquamation had just taken place before opening up or not; but an oil passing through a pitchy condition before coking would give much coke in any case. The high viscosity of the oils chosen is interesting to English chemists, while the heavy consumption of oil 130 m.p.g. would interest commercial users. The quantity of oil passing through the engine is very large, and it is to be expected that a large amount of unused oil would be pumped through to the exhaust manifold, there to be built up into coke formations.

In the absence of exact knowledge of the physical and chemical properties on which lubrication depends, it is necessary to proceed with empirical methods. Much work on the practical side has been wasted owing to bad choice of the oils tested. To gain the greatest amount of useful information in the shortest time, and with the least amount of practical testing, it is important that the *producer* first study the particular question under survey and so refine his oils that as far as possible his products are in the best condition for the work. Trials are best made on a type or crude basis, so that the characteristic behaviour of, say, Pennsylvanian, Mid-Continent, light naphthenic and heavy naphthenic oils is demonstrated and the oils graded as to suitability or length of life. Other types and blended oils may follow.

The best attempt along these lines has been made by Dr. Baader, Dr. Baum, and F. L. Hana, and some of the results have been published under the head of "Dauerversuche über die Alterung von Dampfturbinen-ölen im Betrieb." by Vereinigung der Elektrizitätswerke E.V., Berlin. The number of types tested is not comprehensive, but deductions of considerable value can be drawn. Turbines of roughly 3,000 to 20,000 K.W. on similar duty were used and the chief properties and performance of the oils employed are summarised in Table IV.

The net result of this is to indicate that in practice suitably prepared Pennsylvanian and Russian oils have by far the longest life. The conclusions drawn by the authors were as follows:—

1. The colour gives no indication of the probable life of an oil, or the amount of ageing which has occurred.
2. The specific gravity, flash-point and setting point have little significance as signs of ageing.
3. Refers to water and mechanical impurities and is irrelevant.
4. The appearance of asphalt is always a sign of excessive ageing.
5. Demulsibility *per se* gives no indication of probable life.

TABLE IV.

Oil.	Source.	Refining Process.	Spec. Grav.	O.F.P. ° C.	Engler Viscosity.		
					40° C.	50° C.	70° C.
1	Mexican	Edeleanu and Bleach. Earth.	898	218	7·24	4·77	2·18
2	,,	H_2SO_4 and B.E.	929	206	6·95	4·78	2·06
3	German	,, ,,	913	191	4·96	3·31	2·00
4	S. American	,, ,,	897	204	5·1	3·56	2·0
5	Russian	,, ,,	892	197	4·5	3·17	2·0
6	,,	,, ,,	891	183	4·8	3·41	2·04
7	,,	,, ,,	897	198	4·4	3·04	2·0
8	Penna.	,, ,,	895	206	5·1	3·51	2·17
9	American	H_2SO_4, Alkali and B. Earth	883	212	3·8	2·8	1·85
10	,,	,, ,,	8835	214	3·35	2·7	1·82
11	Puro Oil	H_2SO_4	915	208	5·0	3·75	2·05
12	American	—	904	210	5·8	3·15	1·80
13	,,	—	912	227	7·25	4·7	2·4
14	Russian	H_2SO_4 & Alkali	897	192	4·6	3·05	1·85
15	N. American	Fractionated and An. Charcoal	878	217	4·5	3·15	1·95
16	,,	H_2SO_4, Alkali and B. Earth	883	204	3·65	2·75	1·78
17	American	,, ,,	881	212	3·93	2·80	1·85
18	,,	H_2SO_4 & B.E.	909	190	3·91	2·65	1·80
19	Penna.	—	862	188	4·0	2·80	1·80

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Set. Pt.	Sap. Value.	H_2SO_4 Vol. Decrease.	Increase Sap. Val. 1000 hrs.	
—6	0·86	22·4%	0·26	Still satisfactory after 16,000 hrs.
Below —15	0·35	17·1	0·48	Test discontinued after 8,494 hrs. owing to defective bearing.
—	0·99	18·0	2·35	Oil unsatisfactory after 2,000 hrs.
—7	0·84	25·0	1·72	Oil unsatisfactory after 3,700 hrs.
—14	0·5	2·9	0·2	Still satisfactory after 6,000 hrs. and slightly better than No. 6.
Below —18	0·6	2·8	0·44	Still satisfactory after 6,000 hrs.
—14	0·33	5·0	0·065	Still satisfactory after 21,000 hrs. and one of the best.
—9	—	13·0	0·08	Oil had to be changed after 20,000 hrs.
—8	0·0	5·3	0·20	Still satisfactory after 22,000 hrs.
—9	0·06	7·3	0·09	Still satisfactory after 22,000 hrs.
—10	0·52	10·8	0·25	Test running at 10,000 hrs.
—4	0·59	12·8	0·71	Test stopped at 13,000 hrs. because oil was then unsatisfactory.
—1	0·6	14·5	0·60	Oil unsatisfactory after 8,000 hrs.
—	0·22	5·0	0·06	Test stopped owing to mechanical defect after 5000. This oil really one of the best.
—6	0·53	7·0	0·76	Satisfactory after 13,000.
—	0·41	5·0	0·23	Discontinued after 5000, owing to excessive ageing accelerated by pervious oil cooler.
—	0·22	10·0	0·35	Discontinued after 10,000 for reason above (16).
—15	0·17	7·8	0·36	Oil still satisfactory after 8000 hrs.
—	0·14	10·3	0·28	Oil still satisfactory after 12,000 hrs.

NOTE.—It is well known that practically all types of hydrocarbons can be prepared in an easily demulsifying condition, but that this state is lost sometimes quickly, sometimes after a lapse of considerable time according to the chemical nature of the crude from which the oil is obtained. A good oil will have the characteristics of a recognised good type and a good demulsibility in addition.

6. As tests of ageing during use the only quantitative determinations of any utility are the acidity and the sap. value.

NOTE.—It is later mentioned that comparative tests made on the diminution in volume produced by shaking with sulphuric acid gave a useful indication as to the extent of ageing of a particular oil.

7. The Kissling Tar Value was more difficult to determine and not so useful as the saponification value.

8. The standard oxidation tests did not give reliable indication of length of service.

9. Variations in bearing temperature were in no simple relation to changes in the oil.

NOTE.—One of the chief indications that a machine must shut down is the repeated dangerous rise in the temperature of the bearings.

10. Oil consumption is no measure of the value of an oil or its rate of ageing. Loss of oil is mechanical.

11. Up to the time of the report no chemical test had been evolved which would indicate when the time had come for changing the oil, nor upon which a new oil could be chosen to give satisfactory performance in use.

Some of the trials are still running and Dr. Baader, of the Goldenberg-Werk, Knapsack/köln, has attempted to formulate a test for new oils which will give results in accordance with the length of life of the oils used on the trials. He has worked out an oxidation test based on the determination of the acid and saponification values obtained when the oil is heated 48 hours in the presence of (1) glass, (2) iron, (3) lead, and (4) copper spirals, which are moved in order to circulate the oil. The heating is done on separate samples, each one with one of the above spirals as catalyst. The number obtained in the presence of the glass spiral is called the temperature sensitivity and is deducted from the increase obtained from each of the other tests and so gives what he calls the iron, lead and copper sensitivities. He finds that an oil may be lead sensitive and less sensitive to copper, but generally the reverse is true. The air supply consists of such air as can circulate down the tube of a reflux condenser. Full particulars of the method of testing are in Elektrizitätswirkschaft Nos. 461 and 463, 1928.

Of the oils run in the turbine the factor of ageing taken was the increase in saponification value per thousand hours of running. This number was invariably larger for the short-lived oils.

Oils.	Increase in Sap. Value per thousand hours.	Sap. Value on Test with Copper Catalyst.
Russian H.	0·13	0·06
„ D.	0·15	0·05
„ S.	Under 0·26	0·09
Mexican B.	0·26	0·3
„ A.	0·48	0·69

It will probably be found that the test with copper catalyst alone is sufficient. He recommends that a limit of 0·01 for the acid value and 0·15 for the saponification value be fixed. The suggested method is open to the criticism that the quantities of acid and of saponifiable matter to be estimated are too near the border line of experimental error, and it would be better to have larger numbers with which to work. It will be noted that the method was not drawn up on the whole of the oils tested practically; the reason given for this is that at the time of developing the laboratory tests some of these oils were no longer available. More trials on a larger range of oils will be needed before definite rules can be laid down. The series tested is very useful as far as it goes.

As applied to Transformer oils the figures given by Dr. Baader cannot be made much use of as the type of oil is not always specified and the results, either in practice or by other methods of testing are not known. He gives figures for five other turbine oils and ten transformer oils.

A B.E.S.A. Panel is drawing up a specification for turbine oils, and has wisely decided to introduce an oxidation test. Experience has shown that the Michie Sludge Test is as useful and reliable as any other proposed, and has been adopted at present without any modification. Time is necessary to see whether it would be advisable to alter it in details such as the lowering of the temperature or the estimation of total acidity and saponifiable matter instead of weighing the sludge.

This would appear to be a suitable point at which to draw the attention of enginers to the inadvisability of having bare copper surfaces in the oil circulating system of any engine, or wherever oil has to remain in use for any considerable time. Copper greatly accelerates the initial deterioration of the oil, and rapidly increases the rate of deterioration afterwards. Copper may be tinned, or a metal such as stainless steel substituted.

Ageing test for Transformer Oils.—The B.E.S.A. section of the I.E.C. is continuing its researches on the comparison of sludge

tests, but as the work is not yet published no account can be given.

It may be asked in all seriousness whether anything is gained in the English specification by the very low sludge percentages allowed. We appear to be alone in insisting on low sludge numbers and ignoring the accompanying increase of saponifiable matter. Moreover, the more recent work of the B.E.S.A. in the way of specifying closely the petroleum ether to be used, and following rigorously the method of working, has tended to increase the percentages obtained. Without disadavantge the sludge number for A Grade can be increased to 0·2 per cent., and that for B grades to 1·1 per cent. The instructions for carrying out the B.E.S.A. method of precipitating the sludge neither include mention as to whether the beaker may be allowed to stay in the light nor state at what temperature it should be kept while standing.

Cases have occurred which prove that light increases the amount of precipitated sludge, and that at times, particularly in A grades, a relatively large increase in deposited matter may be obtained at low temperatures. The following is taken from a paper by M. Thiry, and illustrates the danger of low temperature of settling. An A grade oil was placed in a flask and air bubbled through at the temperature of 150° C. until sludge just began to appear. This was cooled and filtered off, and the oil divided into four parts. The same quantity of the same petroleum ether was added to each. Sample (1) was allowed to stand in the light at approximately 20° C.; (2) was placed in the dark at the same temperature. No. 1 gave 0·167 per cent., and No. 2 0·148 per cent. The other two samples were placed in the dark, No. 3 at summer temperature of last year (40° C.), and the other at common winter temperature, average 9° C. (No. 4). No. 3 gave 0·046 per cent., and No. 4 gave 0·3 per cent. sludge.

Carbon Residue Estimation.—C. I. Kelly (*J. Inst. Petr. Techn.*, 1929, **15**, 495) gives a useful demonstration of the utility and accuracy of the Ramsbottom method of taking coke tests. We can with confidence drop the Conradson method.

Other useful pieces of work of this type would be an exhaustive series of Air Board Blowing Factors on typical oils, and the subsequent coke numbers. The A.S.T.M. Pour Point method is also a very unsatisfactory test, and the publication of a fairly complete series of I.P.T. Setting Points compared with A.S.T.M. Pour Points on the same oils would help to clear away a frequent cause of dispute.

The Author wishes to acknowledge his indebtedness to Dr. Ion Edeleanu for information on the process which bears his name, to Mr. J. Kewley for several references, and to my assistant, E. R. Styles, M.Sc., Ph.D., for summarising many references and making translations from the German.

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[11] E.P. 295,230, 1928. H. L. Callendar, R. O. King and J. Mardles.
E.P. 289,909, 1928. F. Hofmann and Dunkel. 262,107.

[12] *Ind. and Eng. Chem.*, August, 1929. W. F. Seyer and J. S. Allen.

Heavy Distillates, Fuel Oils, Asphalts and Residues.*

By F. H. Garner, Ph.D., M.Sc., F.I.C.

The question of specifications of Diesel fuel oil was raised by G. H. Michler in a paper read before the American Society of Mechanical Engineers in 1928.[1]

In this paper, the various specification requirements encountered by the oil refiners are given, and the impossibility of carrying several grades of Diesel oil at distributing points which are not also refining centres was pointed out.

Unless Diesel engines are constructed which can operate efficiently on a standard grade of oil of broad specifications, ship operators will be unduly restricted to limited points of fuel supply and may be excluded from operating in the most desirable trade services.

An American committee has been appointed, consisting of representatives from oil engine manufacturers, oil producers, and this committee has put forward the following specifications for Diesel fuel oil :—[2]

	Heavy Duty Engines.	Light High Speed.
Viscosity @ 100° F. (Saybolt Universal)	Max. 200 sec.	Min. 45 sec. max. 100.
Sulphur	„ 3% by weight	Max. 2%.
Conradson	„ 5%	„ 1%.
Ash	„ 0·08%	„ 0·02%.
Flash	Min. 150° F.	Min. 150° F.
Moisture and sediment ..	Max. 1%	Max. ·5%.

Informative.—Specific gravity or API Gravity is of no importance as pertains to the burning qualities of an oil. It should be given, however, by the supplier in order that weight per gallon can be computed. Pour Test (Local Importance). In localities where low temperatures are experienced, arrangements for suitable fluidity should be made between purchaser and seller.

Mayo D. Hersey[3] investigated the importance of the effect of high pressure on the viscosity of fuel oils to be injected into Diesel engine cylinders, and found that with an oil at 22·6°C. the absolute viscosity rose from 0·0563 Poise at atmospheric pressure to 0·284 at 12,000 lbs. pressure.

The question of gas oil has received a considerable amount of attention during the past two years, and two important articles by R. H. Griffith have appeared.[4] A method is given for the analysis of gas oil into the four groups :—unsaturated, aromatic, naphthene and paraffin, and the relation between the analysis of

* Paper received March 22, 1930.

[1] *Trans. Am. Soc. Mech. Eng.*, 1928, **50**, O.G.P., 50–10.
[2] *Mechanical Engineering—Am. Soc. Mech. Eng.*, 1929, **51**, 765.
[3] *Chem. Abs.*, 1929, **23**, 5566.
[4] *J.S.C.I.*, 1928, **47**, 211T ; 1929, **48**, 252T.

gas oils and the value for the conversion of petroleum fractions into permanent gas is discussed as a result of a considerable amount of experimental work.

The influence of the rate of oil input and temperature changes on the efficiency of gas making were also investigated, together with the quantity and quality of tar produced.

It is concluded that aromatic and naphthene hydrocarbons are of less value than those with straight chain structures, and accordingly the method of valuation is based on the sum of paraffins and unsaturateds.

In a paper by T. A. Mighill[5] the valuation of gas oil is also discussed, and this author places the relative importance of the various types of hydrocarbons as follows:—Assuming paraffin hydrocarbons as 100 per cent., naphthenes rank at 70, aromatic hydrocarbons at 50, and unsaturated at 25.

The method of analysis adopted in this instance for the determination of unsaturated hydrocarbons, consists in treatment of the oil with acid and by centrifuging, separating the unattacked oil. This method seems open to suspicion, as treatment with acid will undoubtedly remove part of the aromatic hydrocarbons.

The Russian standards for Diesel oil, accepted by the Commission for Diesel Oils, are given by N. I. Chernozhukov.[6]

A description of the Hakol-Zwicky process for the manufacture of gas from oil is given in the *Petroleum Times*.[7] A description is given of a modification whereby the difficulty of carbon deposition is overcome, giving a clean and stable gas, which can take the place of town gas for industrial purposes.

The relationship between calorific value and the other characteristics of residual fuel oils and cracked residuum are given by W. F. Faragher, J. C. Morrell and J. L. Essex.[8] For dry oil, the calorific value per pound is given as $17{,}010+(90\times\text{API gravity})$ for straight-run fuel oils, and $17{,}645+(54\times\text{API gravity})$ for cracked residuums.

As a result of the conference held under the auspices of the United States Bureau of Standards, recommendations for furnace and fuel oil standards are given, covering the requirements of oils both for domestic burners and industrial use.[9]

An interesting paper on road emulsions has been published by P. E. Spielmann.[10] In this paper the relative merits of emulsions, tar and hard bitumen are discussed.

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[5] *Chem. Abs.*, 1928, **22**, 4783.
[6] *Chem. Abs.*, 1929, **23**, 4810.
[7] *Petroleum Times*, **18**, 1192; *J. Inst. Petr. Techn.*, 1928, **14**, 17A.
[8] *J. Ind. Eng. Chem.*, 1929, **21**, 933, 1287.
[9] *J. Inst. Petr. Techn.*, 1929, **15**, 127A.
[10] *Roads and Road Construction*, 1930, 10.

Specifications of the emulsions manufactured from tar and bitumen have been drawn up with a view to submission to the British Engineering Standards Association.[11]

A number of patents have been taken out in connection with the manufacture of emulsions from bitumen.

C. H. Thompson and W. J. McGivern have patented the use of liquid rosin derived from sulphite cellulose together with a natural bitumen and an alkali such as caustic soda.[12]

F. Levy describes the use of Mexican or other asphalt in molten condition, which is emulsified with a hot aqueous solution of alkaline silicate without the use of fatty substances.[13]

H. Plauson[14] has described the use of bitumen emulsion produced by emulsifying a glyceride and potassium carbonate, and then mixing the bitumen with the emulsion so formed.

J. A. Montgomerie[15] uses hot aqueous solution containing normal sodium phosphate or other suitable tertiary phosphate in proportions of about 0·5 per cent. to that of the bitumen.

The B.E.S.A. have issued a new series of specifications dealing with the use of asphalt for road purposes.

W. Wilhelmi[16] discusses the essential properties of asphalt and tar emulsions, and gives his views as to the types of bitumens and tars which should be used in their manufacture. He also gives brief descriptions of a number of commercial emulsions.

H. Burstin[17] gives the paraffin wax contents of three asphalts as determined by the Schwarz, Marcusson and cracking methods, and concludes that, of these various methods, the Marcusson is the most accurate.

A. N. Sakhanov[18] has carried out experimental work on the solidification of paraffin containing residues derived from Grozny crude and mazout, and finds that the solidifying point varies not only with the temperature to which the oil is being heated, but that the highest solidification point corresponds to a pre-heating temperature which lies between 40-60°C., but varies with the paraffin content. Higher pre-heating temperatures cause a depression of the solidification point, this depression disappearing on standing.

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[11] *The Contractors Record & Municipal Engineering*, 1929, **40**, 1543.
[12] E.P. 263,307, *Chem. Abs.*, 1928, **22**, 163.
[13] E.P. 268,411. *Chem. Abs.*, 1928, **22**, 1471.
[14] Can. P. 276,480. *Chem. Abs.*, 1928, **22**, 2054.
[15] E.P. 300,414. *Chem. Abs.*, 1929, **23**, 4061.
[16] *Erdöl und Teer*, 1929, **5**, 87, 105.
[17] *Chem. Abs.*, 1929, **23**, 2563.
[18] *Chem. Abs.*, 1929, **23**, 269.

Special Products.*

By J. A. Carpenter, M.A., F.I.C. (Member).

Paraffin Wax.

Crystallisation and Structure.—An interesting contribution by L. D. Myers and G. Stegeman[1] deals with the formation of solid solutions of paraffin waxes in one another. These solutions possess properties intermediate between those of their constituents, even with regard to changes of their solubilities in oil about 10° C. below their melting-points. Oil also can be retained in the form of a solid solution, and the last 2 per cent. is exceedingly difficult to dislodge even at 3000 lb. per sq. in. pressure.

G. A. Burrell[2] has published a useful résumé on the composition and properties of paraffin wax, in which he has reviewed recent work on crystallisation and on the controversy regarding the identity of crystalline and amorphous waxes.

F. Padgett[3] (also Morritt and Padgett)[4] considers that waxes in petroleum distillates crystallise in the orthorhombic system, and that the same is probably true of wax in residues, though the crystal habit varies in the different products. While distillate wax may take the form of leaflets, plates, needles or fibres, that in the residues is commonly termed amorphous, though probably it exists as crystals which are too small in size and soft in consistency to be recognised as such. The habit and consistency are influenced by impurities during the crystallisation; this view is more likely than that in which amorphous branched-chain paraffins are supposed to occur in residues becoming isomerised to normal crystalline paraffins on distillation.

E. Pyhala[5] has found that the Scotch method of dry sweating crude paraffin wax is successful only if the crystals have the needle form.

A further valuable contribution is that by Y. Tanaka, R. Kobayashi, and S. Ohno[6], who have examined paraffin from shale-oil, also from Echigo, Formosa and Californian petroleums.

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* Paper received December 30, 1929.

[1] *Ind. Eng. Chem.*, 1928, **20**, 638.

[2] *Oil and Gas J.*, October 18, 1928, 130.

[3] *Oil and Gas J.*, March 7, 1929, 248.

[4] Paper before Amer. Chem. Soc., Swampscott, September, 1928.

[5] *Chem. Abstr.*, 1929, **23**, 3076.

[6] *J. Fac. Eng., Tokyo*, 1928, **17**, 275 and *J. Inst. Petr. Techn.*, 1929, **15**, 74A. Also *Rep. Imp. Fuel Res. Inst. Japan*, No. 8 (1929); *Chem. Abstr.*, 1929, **23**, 5036.

The greater portion of shale paraffin consists of normal hydrocarbons, crystallising from its more concentrated solutions in alcohol in needles and from weaker solutions in plate forms, the latter being preferentially formed also under slow cooling conditions. Differences in crystalline type depend on the cooling velocity and on the viscosity of the medium, not on the chemical nature of the paraffin.

Low viscosity of medium favours formation of plates; high viscosity that of needles; while petroleum and shale paraffins are, range for range, identical in all respects.

The simple form of crystal is a rhombic plate having angles of 110° and 70° exhibiting double refraction and parallel extinction. The best results are obtained from highest dilution and lowest cooling velocities; complex plate forms are considered to be produced by parallel growths of the rhomb in the direction of the 110° angle, while needle forms grow similarly in the direction of the 70° angle and show the same optical properties as plates though they differ in angle of optic axes. Needle forms can be obtained from plates by recrystallisation from a viscous oil, while the reverse effect can be obtained through a light non-viscous oil. The disturbance to crystallisation from low-temperature tar by the presence of amorphous impurity is also discussed and an equation is given correlating the velocity of crystal growth with amount of impurity, rate of cooling, viscosity, concentration and solubility.

Peck in a paper before the American Chemical Society (September, 1928) has found that good "pressable" wax crystallises uniformly from its distillate, whereas poor wax may appear in various forms, depending on the temperature.

L. D. Jones and F. E. Blachly[7] have published work on the properties of crystalline and amorphous wax as well as the various conditions whereby wax is precipitated in the crystalline or amorphous state. They consider that the condition is largely a function of surface tension affected by surface adsorption of some impurity—possibly the soft wax of C. C. Buchler and G. D. Graves[8] or some other constituent. Addition of amorphous material to a solution from which good crystals of wax can be precipitated without such addition is capable of rendering the whole precipitate amorphous. Adsorption takes place more easily at low temperatures, for at higher temperatures the amorphous substances are in true solution in the accompanying oil.

In reviewing the above papers in conjunction with the previous work of Padgett, Hefley and Henriksen, Carpenter, Buchler and

[7] *Ind. Eng. Chem.*, 1929, **21**, 318.
[8] *Ind. Eng. Chem.*, 1927, **19**, 718.

Graves, Rhodes, Mason and Sutton, Sachanen and Bestuchen (see Burrell's paper mentioned above or previous naphthology progress reports for references) together with X-ray studies mentioned below, it is seen that much light has been thrown on knowledge of the habits of waxes.

Whatever minor discrepancies may appear between the views of some of the authors, it is agreed between all that plates represent the most stable form for the higher waxes at ordinary temperatures, and that differences in crystal size or type depend on the velocity of cooling, viscosity of the medium, and presence of amorphous impurities.

Control of the temperature ranges in relation to crystal habits with reference to viscosity of the medium for both solutions and pure waxes may yet repay further study, including regulated velocities of cooling in upper and lower ranges of temperature.

The work of Tanaka and his colleagues brings many views into line with each other, though the exact nature of the influence of soft impurities on crystalline habit as noted by Buchler and Graves, together with observations on the curling of plates into apparent needles as noted by Rhodes, Mason and Sutton, have not yet been correlated fully with, for example, Tanaka's observations on the method of growth of rhombs when assuming the needle form.

The modern view of the identity of crystalline wax with that already present in solution in amorphous residues is again exemplified by the work of Jones and Blachly following that of Gurwitsch[9] and many of the authors noted above, all of whom have now demonstrated in practically the same manner that there is no need for the older view of Zaloziecki, Engler, Boehm, Marcusson and others who regarded amorphous residues as containing amorphous iso-paraffins transformed by distillation into crystalline normal paraffins. The ability to obtain good crystals of "pressable" wax depends fundamentally not on such transformations (even though cracking of associated substances may take place simultaneously in some minor degree giving liquid or less amorphous distillates which no longer hinder the crystallisation) but on the efficiency of separation of the crystallisable wax from amorphous material by good fractionation or any other physical or chemical method which removes the amorphous material from the sphere of action.

As an example of the general application of this principle in refining practice, A. G. Peterkin[10], in comparing tube still practice with coking still and steam running, has shown that the good

[9] "The Scientific Principles of Petroleum Technology" (Trans. Moore) p. 255.

[10] *Oil and Gas J.*, January 5, 1928, 64, 101.

fractionation obtained by a tube still-bubble tower plant enables a higher yield of wax distillate to be taken off with cleaner separation from high-boiling amorphous material, resulting in perfect ease of sweating and pressing while practically no cracking has taken place. A very sharp demarcation of "pressable" quality was noted in one case corresponding to the temperature at which the last 5 per cent. boiled.

Buchler and Graves have demonstrated the same thing, while A. Ablovatzkii[11] has referred to the same principle in Russian practice.

The full chemical and physical characteristics of the high-boiling or truly amorphous jellies are still open to question. A good discussion of this (and indeed of all wax subjects) appears in "Petroleum and its Products" (1928), by W. A. Gruse. It may be noted that in many citations on this subject, the apparent discrepancies of views as suggested therein disappear entirely when it is remembered that two different materials have been under review. On the one hand a solution of crystallisable wax in amorphous material, and on the other hand the residual amorphous material itself, after the crystallisable wax has been removed, both of which have been often referred to as "amorphous jelly" or "amorphous wax." While the latter viscous or semi-solid hydrocarbons may yet retain and mask some measure of paraffins, Gruse (*loc. cit.*) in his summary considers them to be probably of a cyclic nature.

Among papers dealing with the chemical characteristics of mineral jellies, the following have appeared :—

R. Poggi (*Giorn. Chim. Ind. Appl.*, 1928, **10**, 601 ; *Brit. Chem. Abstr. B*, 1929, 159). Determination of unsaturation in mineral jellies for the manufacture of cordite and comparisons of Rosenmund with Kuhnheim bromine or iodine value methods.

A. S. Velikovsky and S. S. Nifontava (*Chem. Abstr.*, 1929, **23**, 4332). Comparison of Russian and American Mineral Jellies.

P. Agostini (*Ann. Chim. Appl.*, 1929, **19**, 241. *Brit. Chem. Abstr., B*, 1929, 768). Comparisons of McIlhiney and Rosenmund Methods for unsaturation.

Chemical Constituents of Paraffin Wax.—S. W. Ferris, H. C. Cowles and L. M. Henderson (*Ind. Eng. Chem.*, 1929, **21**, 1090) have cut oil-free waxes by vacuum distillation and, by refractionations from ethylene dichloride, have succeeded in producing purified waxes of similar molecular weights and boiling ranges, but with widely differing solubilities, melting points and refractive indices. Their results confirm clearly that isomers, probably of a saturated branching chain nature and not of an unsaturated or cyclic character, are present in addition to normal straight chain hydrocarbons in solid petroleum waxes.

[11] *Chem. Abstr.*, 1929, **23**, 4813.

X-Ray Studies.—Increasing attention has been devoted to X-ray methods of structure analysis chiefly following the work which has developed during the last few years on the determination of spacings from X-ray powder diagrams.

G. W. Stewart[12] has examined X-ray diffractions of normal paraffins up to pentadecane.

J. Hengstenberg[13] has found that $C_{35}H_{72}$ is orthorhombic with two mols. in the unit cell of dimensions, a=7·43, b=4·97, c=46·2 Å.U. The CH_2 groups are spaced in the direction of the C axis, 1·27 Å.U. apart.

A mixture of high molecular weight paraffins shows the same simple CH_2 groups; a zig-zag arrangement of CH_2 groups in a molecule is suggested.

A. Muller[14] has purified $C_{29}H_{60}$ and checked its composition against pure synthetic $C_{30}H_{62}$ and $C_{31}H_{64}$ by comparing X-ray powder diagrams. A single small crystal mounted on a glass fibre is placed in the goniometer of an X-ray spectrograph so that complete rotation and oscillation photographs can be taken with copper radiation. The crystal is of the orthorhombic space group Q_h^{16}; many measurements are given in the paper regarding zig-zag arrangements and other matters, while the unit cell is noted as a=7·45, b=4·97, c=77·2 Å.U.

G. L. Clark[15] has summarised data for paraffin waxes, giving detailed X-ray diffraction data on normal hydrocarbons, both in the crystalline and liquid states.

In thin layers, molecules arrange themselves parallel to each other, forming equidistant layers, the spacing of which is a measure of molecular weight and bears a linear relation to the number of carbon atoms. Variations in crystalline form are noted for lower hydrocarbons, but in liquids no diffraction maxima are found as with alcohols, acids and other compounds with polar and groups. Further notes are given on the effect of other bodies, such as acids, soaps, dyes and bases, on the variation of the spacings.

An interesting point brought out is that paraffin wax, though composed of mixed normal hydrocarbons, yields diffraction line, characteristic of only one spacing depending on a pure components even though that component may be present in small quantity.

The spacing is a function of melting-point, rate of crystallisation, source of wax and other variables, but is by no means evenly progressive for narrow cuts. This paper, which is intended to stimulate research, should be consulted, when suggestions regarding its

[12] *Phys. Rev.*, 1928, **31**, 174; *Chem. Abstr.*, 1928, **22**, 1098.
[13] *Z. Krist.*, 1928, **67**, 583; *Chem. Abstr.*, 1928, **22**, 4289.
[14] *Proc. Roy. Soc.*, 1928, **A-120**, 437; *Chem. Abstr.*, 1929, **23**, 30.
[15] Paper before Amer. Chem. Soc., Swampscott, September, 1928.

connexion with other work (particularly that on solid solutions by Myers and Stegeman, also the conclusions of S. H. Piper, D. Brown and S. Dyment[16] from X-ray studies) will present themselves to the reader.

In this connexion a new universal X-ray photogoniometer is described by J. D. Bernal[17].

The general discussion by the Faraday Society (published March, 1929) on "Crystal Structure and Chemical Constitution," including Prof. V. M. Goldschmidt's lecture, provides a valuable review of X-ray technique in crystal studies.

Physical Constants.—P. Weber and H. L. Dunlap[18] have determined the solubilities of pure solid paraffin in pure low-boiling paraffins. They find that the solubility rises rapidly with rise of temperature, and conclude, on the strength of a few results, that the molar ratios of solvent to solute are constant—*i.e.*, in each case definite numbers of molecules of solvent and solute are associated with each other.

O. G. Wilson[19] has given charts for the determination of vapour pressures of normal paraffins up to C_{35} H_{72}.

J. A. Carpenter[20] has isolated from petroleum the complete range of paraffins from C_7 to C_{32} in a fair degree of purity by fractional processes of various kinds following the destruction of other classes of hydrocarbons, or by pyrolysis of higher paraffins followed by similar treatment. Boiling-points under various pressures, aniline points, melting-points and some refractive indices are given.

S. T. Minchin and G. R. Nixon[21] have dealt with the pyrolysis of wax with a view to obtaining the physical constants of lower paraffins obtained thereby as reference standards for spirits and kerosines. They have also compiled useful tables of constants for a great variety of paraffin and other hydrocarbons.

G. S. Parks and S. S. Todd[22] have determined the heats of fusion and specific heats of paraffins by dropping capsules of hydrocarbons at various temperatures into cold water. They develop a formula for the heat of fusion based on molal entropies, but observe that branched chain paraffins show irregular results although the entropy of fusion apparently decreases with increase of the amount of branching. (See also reference 52.)

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[16] *J. C. S.*, 1925, **127**, 2194.

[17] *J. Sci. Instruments*, 1929, **6**, 314, 343.

[18] *Ind. Eng. Chem.*, 1928, **20**, 383.

[19] *Ind. Eng. Chem.*, 1928, **20**, 1363.

[20] *J. Inst. Petr. Techn.*, 1928, **14**, 446.

[21] *J. Inst. Petr. Techn.*, 1928, **14**, 477.

[22] *Oil and Gas. J.*, September 26, 1929, 143 (Paper before Amer. Chem. Soc., Minneapolis). *Ind. Eng. Chem.*, 1929, **21**, 1235.

The properties of paraffins are also touched upon in various papers dealing with pyrolysis or analysis of hydrocarbon classes—*e.g.*, R. H. Griffith (*J.S.C.I.*, 1928, **47**, 21T), F. H. Garner (*J. Inst. Petr. Techn.*, 1928, **14**, 695), J. H. Hildebrand and A. Wachter (*J. Amer. Chem. Soc.*, 1929, **51**, 2487) and others.

Pyrolysis and Oxidation.—H. I. Waterman, J. N. J. Perquin, and H. A. Van Westen[23] have continued their work on the cracking and berginisation of Rangoon wax. Paraffin wax is relatively thermo-resistant up to temperatures of 370° C., but heating for ten hours at 370° C. leads to a lowering in average molecular weight from 410 to 360 while the gases, low-boiling distillates and residues contain unsaturated hydrocarbons.

H. I. Waterman and L. L. W. Van Soest[24] heated Rangoon wax of M.Wt. 414 with aluminium chloride at temperatures gradually increasing in successive experiments to 300° C. under vacuum. Light saturated gasoline began to form at 180° C., but the action of the aluminium chloride was limited, and possibly some of the effect on the residues was produced by simple distillation after the $AlCl_3$ had become inactive. During the period of activity at the initial lower temperatures, saturated products were probably formed by the condensing action of $AlCl_3$ on nascent unsaturateds, as found by other experimenters.

H. I. Waterman, T. W. de Nuyl, and J. N. J. Perquin[25] compared the cracking and berginisation of wax, from which it was found that the properties of the berginised products were practically those of normal saturated paraffins, while those of cracked products corresponded to a mixture of saturated, unsaturated and cyclic derivatives.

J. A. Carpenter[20], also S. T. Minchin and G. R. Nixon[21] have observed that liquid-phase cracking (as opposed to vapour-phase or other conditions specially suited to the formation of cyclic bodies) of higher paraffin waxes gives high yields of lower saturated paraffins together with aromatics and unsaturateds, but very small yields of naphthenes and practically no coke. The first named has also observed that there are indications of the formation of naphthenes and possibly of saturated paraffins by sulphuric acid treatment of the cracked products through action on some of the unsaturateds, a result in line with the observation of others.

A. N. Sachanen and M. D. Tilitscheev[26] have continued their comprehensive work on the cracking conditions of many classes of

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[23] *J. Inst. Petr. Techn.*, 1928, **14**, 318.
[24] *J. Inst. Petr. Techn.*, 1928, **14**, 756.
[25] *J. Inst. Petr. Techn.*, 1929, **15**, 369.
[26] *Ber.*, 1929, **62**, 658, and *Brit. Chem. Abstr.* B, 1929, 383. *Chem. Abstr.*, 1929, **23**, 3658; also *J. Inst. Petr. Techn.*, 1929, **15**, 294A.

hydrocarbons, including paraffin wax. Paraffins crack in the first instance to unsaturateds and lower paraffins; unless these are removed quickly from the sphere of action, the unsaturateds polymerise to aromatics with naphthenes, and then possibly begin to form coke if the action is prolonged, particularly under high pressures. Paraffins and cyclic hydrocarbons with long side chains are more unstable than condensed nuclear bodies with short side chains, so that the rate of decomposition falls when such conditions are established. This work seems to bear on certain observations by others regarding the predominance of aromatics with short alkyl side chains in certain types of crude oil as produced by nature.

V. Ipatiev and N. Orlov[27] have obtained similar results with hydrogen and alumina. R. H. McKee and H. H. Parker[28] have studied critical temperatures as a basis for differentiation between liquid and vapour-phase cracking; for saturated aliphatic straight or branched chain hydrocarbons they deduce the formula:— t_c: 1·05 t_b+160 where t_c=critical temperature in Centigrade degrees and t_b=average boiling-point on an A.S.T.M. distillation basis.

F. Hebler[29] has collected information on the oxidation of paraffins, including wax, to fatty acids and other bodies.

The I.G. Farbenindustrie A.G., through J. Y. Johnson[30] has taken out patents on the oxidation of paraffin waxes. In the first it is noted that oxidation by air in presence of inorganic catalysts is accelerated by the presence of organic nitrogenous bases. In the second, recovery of lower melting oxidation products is effected by filter pressing or by salt formation, while the third is an improvement of a previous process, and mentions the use of organic salts of alkaline earth, metals, magnesium or aluminium as catalysts. The fourth deals with the removal of oxidised acids through salt formation. The same firm has also taken out E.P. 274,095 for the production of cyclic ketones by condensation.

Processing, Analysis and Behaviour of Wax-Oil Mixtures.—The conclusion of a general article on wax and its manufacture was published by G. W. Cupit[31], while the same author[32] gave further notes in a paper on the processing of coking crudes.

E. R. Lederer and F. R. Staley[33] have discussed the effect of wax

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[27] *Ber.*, 1929, **62**-B, 593, and *Chem. Abstr.*, 1929, **23**, 3697.
[28] *Ind. Eng. Chem.*, 1928, **20**, 1169.
[29] *Erdol und Teer*, 1928, **19**, 333, and *J. Inst. Petr. Techn.*, 1928, **14**, 208A.
[30] E.P. 298 704 of 1927, 309 875, 310 069 and 315 813 of 1929; see *J. Inst. Petr. Techn.*, 1929, **15**, 14A, 187A and 260A.
[31] *Refiner and Nat. Gas. Mfr.*, January, 1928, **7**, No. 1, 67.
[32] *Refiner and Nat. Gas. Mfr.*, March, 1929, **8**, No. 3, 71, and April, 1929, **8**, No. 4, 86.
[33] *Oil and Gas J.*, March 21, 1929, 158.

on aviation engine oils, showing that a reduction of only 0·2 per cent. of wax may cause the temperature of change from plastic liquid to plastic solid to alter from +10° F. to – 10° F., though the difference in A.S.T.M. pour-point is only 2° F.

Variations in the crystalline or amorphous nature of the wax, as well as in the type of accompanying oil make for differences such that the pour point test may give a false idea of the behaviour of the oil.

E. Petty[34], in an article on the handling of vacuum-distilled wax-bearing products has dealt with the physics of the separation of wax at low temperatures and gives curves showing the relation between the volume of the naphtha used for dilution of mixtures and petrolatum precipitation. Wax is gradually precipitated from naphtha solution as the temperature is lowered ; for mixtures containing 60–70 per cent. naphtha there is a crystallisation " peak " about 50° F. to 30° F. This results from the coagulation of very finely dispersed wax particles, but as these particles take a more definite shape at about 30° F., there is a sudden change of solvent arrangement as the wax throws off naphtha and the oil takes up all that is discarded. By continued chilling the first wax gives off naphtha and becomes firm, but towards –40° F. another saturation point is reached and the remaining wax is precipitated. Hence chilling for complete dewaxing is resolved into two distinct cycles.

G. F. Olsen[35] makes use of double centrifuging on the above lines by chilling to 20° F., separating wax, then chilling again. The second slack wax is added to a fresh batch of diluted oil.

P. Truesdell[36] discusses the effects of " forty below zero " chilling on oil as carried out by the direct ammonia expansion method of M. B. Miller, who gives a detailed description[37] of the process in a later article.

Oils dewaxed by this method possess a high degree of fluidity at low temperatures.

P. Truesdell[38] describes practice at the refinery of the Transcontinental Oil Co., who prefer the cold settling method to that of centrifuging.

G. T. Yost[39] describes an improved super centrifuge operating at 15,000–17,000 r.p.m. as used by Pure Oil Co. for the separation of petrolatum from bright stock.

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[34] *Refiner and Nat. Gas Mfr.*, June, 1929, **8**, No. 6, 62.
[35] U.S.P. 1,715,670.
[36] *Nat. Petr. News*, January 23, 1929, **21**, No. 4, 27.
[37] *Nat. Petr. News*, January 30, 1929, **21**, No. 5, 69.
[38] *Nat. Petr. News*, June 26, 1929, **21**, No. 26, 80.
[39] *Oil and Gas J.*, February 21, 1929, 156.

J. W. Weir[40] gives a note on a laboratory apparatus for dewaxing lubricating oil stocks with filter aids and vacuum flash.

M. Morgenstern[41] has produced a new method for the separation of waxes and asphalts from oil residues for which great claims are made. The method operates by direct means with the aid of low-pressure steam.

G. D. Graves[42] removes waxes which hinder filtration or sweating from oil distillates prior to the final dewaxing process by filtration at a temperature at which the ordinary wax remains in suspension, but below that at which solidification of the objectionable waxes occurs.

E. B. Phillips and J. G. Stafford[43] dewax oils by means of filter aids and finely-divided solids in filtration after chilling. W. T. Ziegenhain[44] also discusses the same principle.

F. W. Sullivan, W. J. McGill and H. L. Walker[45] introduce water into the oil cake, while L. W. Naylor[46] uses litharge as a filter aid.

E. B. Cobb and A. Holmes[47] use isopropyl alcohol to remove sulphonates after fuming acid treatment of crude petroleum with subsequent clay treatment. R. S. Lane[48] removes petrolatum from oils by dilution with a mixture containing 35–60 per cent. naphtha, 25–35 per cent. butanol and 12–35 per cent. acetone. The wax is separated on chilling the mixture through differences in specific gravities.

E. R. Wiles[49] uses a solvent composed of 35 per cent. acetone and 65 per cent. benzol. R. E. Wilson[50] uses an alcohol (C_3 to C_7) such as isopropyl alcohol in a similar manner.

References to other methods will be found on pp. 224A and 226A of *J. Inst. Petr. Techn.*, 1928, including an SO_2 method (E.P. 263,167) by the Bataafsche Petr. Maatschappij, and the use of Butanone (or methylethyl ketone), or of mixtures of this or butyl-alcohols with naphtha by C. R. Wagner (in U.S.P. 1,669,151, 1,669,180).

Heavy liquids such as trichlorethylene are used by N. D. Backlund (U.S.P. 1,676,069).

A process which has excited interest in refining circles is that of F. X. Govers[51], a brief account of which is given by G. H. Reid.

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[40] *Oil and Gas J.*, March 7, 1929, 113.
[41] *Petroleum Z.*, February 27, 1929, 3.
[42] U.S.P. 1,668,239.
[43] U.S.P. 1,714,133.
[44] *Oil and Gas J.*, October 3, 1929, 347.
[45] U.S.P. 1,720,300.
[46] U.S.P. 1,722,307.
[47] U.S.P. 1,707,187.
[48] U.S.P. 1,706,120.
[49] U.S.P. 1,684,426.
[50] U.S.P. 1,719,350.
[51] *Chem. Met. Eng.*, 1929, **36**, 400; *Refiner and Nat. Gas. Mfr.*, October, 1929, **8**, No. 10, 63.

A more detailed paper is to be submitted by Prof. Wells about this process, which deals with the use of diphenyl vapour combined with vacuum operations, solvent extraction of paraffin to produce wax-free oils, chemical treatment, filtration and further vacuum distillation.

The advantages of diphenyl, as exhibited by its chemical and physical characteristics, fit in well with its use as an indirect heating medium, thus permitting high efficiency of heat transfer with low-temperature differences and avoidance of local overheating.

J. W. Poole[52] has discussed the possibilities of solvent extraction of wax from oils, and after reviewing the properties of several potential solvents, indicates that their application to commercial work is being studied further.

Luts and Pettai[53] have compared methods for determining wax in asphalts and pitches. As between the Engler Holde, Butanone and I.P.T. Standard Method, they prefer the Butanone method for accuracy and simplicity.

D. P. Weld[54] has compiled a useful summary of methods for the determination of oil in wax with comments on their merits.

Of other work, the following patents or articles are mentioned :—

C. Boesch. E.P. 287,094.
T. Clarkson and H. B. Heal. U.S.P. 1,686,437.
B. L. Souther and W. A. Gruse. U.S.P. 1,685,008 and 1,685,058.
P. M. Travers. U.S.P. 1,732,143/4.
A. Holmes. U.S.P. 1,733,597. Manufacture of White Petrolatum.
J. Y. Johnson and I. G. Farb. A.G. E.P. 320,921. Refining Crude Wax by Hydrogenation.
L. C. Jamieson.[55] Cold Settling.
C. R. McKay.[56] Dewaxing Practice.
H. L. Kauffman.[57] Experimental Sweating of Slack Wax.
N. I. Chernozhukov and B. K. Tarasov.[58] Manufacture of Petrolatum, etc.
G. W. Cupit.[59] Production of Transformer Oils.
E. R. Lederer and R. Beattie.[59a] Dewaxing of Lubricants.

Insecticides.

The use of petroleum or its derived products in insecticides and larvicides has developed considerably during the period under review In the notes given below, no reference has been given to the vast

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[52] *Oil and Gas J.*, September 26, 1929, 99 ; *Ind. Eng. Chem.*, 1929, 1098.
[53] *J. Inst. Petr. Techn.*, 1929, **15**, 151A.
[54] *Oil and Gas J.*, September 26, 1929, 44 ; *Refiner*, October, 1929, **8**, No. 10, 102.
[55] *Refiner and Nat. Gas. Mfr.*, October, 1928, **7**, No. 10, 92.
[56] *Oil and Gas J.*, December 27, 1928, 168.
[57] *Refiner and Nat. Gas. Mfr.*, September, 1929, **8**, No. 9, 74.
[58] *Chem. Abstr.*, 1929, **23**, 4812.
[59] *Refiner and Nat. Gas. Mfr.*, July, 1928, **7**, No. 7, 63.
[59a] *Oil and Gas J.*, December 5, 1929, 115.

amount of work appearing on inorganic poisons such as arsenates, metallic salts and such like, though the references to various substances directly, indirectly or potentially connected with the use of mineral oils are reviewed.

R. T. Cotton and R. C. Roark[60] have found that alkyl and alkylene formates are toxic to insects which affect stored products; fire hazards can be removed by the incorporation of carbon tetra chloride. Chloro derivatives of fatty acids, also ethylene oxide, give good results.

E. R. De Ong[61] has published papers on types of petroleum emulsions used for spraying trees, especially citrus and deciduous trees, with and without other substances such as nicotine.

The use of petroleum oils with high unsulphonated residues is noted, while the effect of persistent fractions on functional disturbances to the trees may be avoided by using volatile fractions.

The same author[62] also finds that sulphur, oxidation and unsaturated products are deleterious to plants, so that oxidation rate (*e.g.*, method of Sligh) and sulphonation tests are useful for giving measures of safety in use. Refined oils are less insecticidal, but this can be compensated by the addition of toxic agents. Refined pine oils are useful.

R. S. Woglum[63] also gives a report on the use of oil sprays in California with notes on the relation between the properties of oils and effects on the trees, also the combination of sprays with fumigants.

C. H. Peet[64] has given a note on the evaluation of insecticides through toxicity trials, while M. Francois and L. Seguin[65] have given chemical methods for the analysis of insecticides containing petroleum and other products.

C. R. Twinn and F. A. Herman[66] have prepared a useful 6 per cent. pyrethrum in oil spray (½lb. to 1 gallon kerosine) subsequently adding methyl salicylate at the rate of three fluid ounces to a gallon. Precautions to avoid exposure during mixing are important owing to the rapid deterioration of pyrethrum on exposure.

Pyrethrum extract (from Chrysanthemum Cinerariaefolium, Coccineum and Marschallii Ascher) has received a great deal of

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[60] *Ind. Eng. Chem.*, 1928, **20**, 380, 512 and 805.

[61] *Ind. Eng. Chem.*, 1928, **20**, 826; *J. Econ. Entomol.* 1928, **21**, 502 and 525; *Chem. Abstr.*, 1928, **22**, 4197, 4708.

[62] *J. Econ. Entomol.*, 1928, **21**, 697; *Chem. Abstr.*, 1929, **23**, 467.

[63] *J. Econ. Entomol.*, 1928, **21**, 530; *Chem. Abstr.*, 1929, **23**, 4708.

[64] *Ind. Eng. Chem.*, 1928, **20**, 1164.

[65] *J. Pharm. Chim.*, 1928, **7**, 331; *Chem. Abstr.*, 1928, **22**, 3255, 3948; 1929, **23**, 4523.

[66] *J. Sci. Agr.*, 1928, **18**, 441; *Chem. Abstr.*, 1928, **22**, 1429.

attention owing to the great extension of its use in liquid insecticides during the last five years.

J. C. F. Fryer, F. Tattersfield and C. T. Gimingham[67] give detailed accounts of the experimental culture of pyrethrum chrysanthemums in England with reference to work in Dalmatia, Switzerland and Japan. Toxicity to insects is found to be high, particularly in extracts from the flowers which possess up to ten times as much lethal property as the stalks. The keeping properties of extracts are also discussed.

W. A. Gersdorff and W. M. Davidson[67a] have carried out work on solvents for extracting the active toxic principle of pyrethrum.

Accounts of the isolation of the active principles, Pyrethrin I and II, following the earlier work of Staudinger and Ruzicka, appear in this paper with further work in papers by F. Tattersfield, R. P. Hobson and C. T. Gimingham[68] who found that Pyrethrin I is ten times as toxic to bean aphis as Pyrethrin II.

Against this, C. B. Gnadinger and C. S. Cori[69] have also isolated these active constituents (which they find in the proportions of 0·4 to 1·21 per cent. in the flowers), but their tables show only a slight advantage of Pyrethrin I over Pyrethrin II in toxicity to cockroaches. They have confirmed that pyrethrum stems contain about one-tenth the amount of pyrethrins found in the poorest flowers, that daisies contain no pyrethrin and that the active principles of Dalmatian and Japanese pyrethrum flowers are identical.

S. W. Frost[70] finds that petroleum oils emulsified with calcium caseinate in the cold show a high toxicity against rosy aphis on apple trees at safe concentrations of about three per cent.

J. M. Ginsburg[71] reaches the conclusion that the most toxic fractions of petroleum for use as mosquito larvicides are those boiling between 350° F. and 740° F. A mixture containing 10 per cent. 300–400° F., 40 per cent. 400–500° F. and 50 per cent. 550–750° F., with a viscosity of 50 seconds Saybolt at 100° F., combines toxicity with sufficient persistence to outlast the life cycle of a larva.

The same author[72] correlates the use of oil sprays with resultant chlorophyll content of foliage, showing that leaves of apple trees sprayed with refined lubricant fractions possess more chlorophyll than untreated leaves.

F. T. Shutt[73] finds that very minute additions of caustic soda

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[67] *Ann. Appl. Biol.*, 1928, **15**, 423; *Chem. Abstr.*, 1928, **22**, 4708.
[67a] *Ind. Eng. Chem.*, 1929, **21**, 1251.
[68] *J. Agr. Sci.*, 1929, **19**, 266, 433; *Nature*, 1929, **124**, 155.
[69] *J. Amer. Chem. Soc.*, 1929, **51**, 3054.
[70] *J. Econ. Entomol.*, 1928, **21**, 504; *Chem. Abstr.*, 1928, **23**, 4197.
[71] *Oil and Gas J.*, December 27, 1928, 150.
[72] *J. Econ. Entomol.*, 1929, **22**, 360; *Brit. Chem. Abstr.*, B, 1929, 831.
[73] *Chem. Abstr.*, 1929, **23**, 466.

solutions in water or alcohol to the spray fluid improve the continuous nature of oil films on water.

The use of organic nitrogen bases, sulphocyanides and such like in weevil sprays is the subject of patents[74] granted to the I. G. Farbenindustrie A. G.

This recalls British Patent No. 287,371 of 1927 to Associated Oil Co. for the use of basic nitrogen compound from sulphuric dioxide extracts from shale-oils.

M. T. Inman[75] has shown that sulphonated oxidation products of petroleum (obtained by means of catalytic vapour phase oxidation to mixtures of alcohols, ketones, acids, etc.) when incorporated with toxic bodies such as nicotine and pyrethrum in petroleum sprays, behave as activators, mainly probably through the increased wetting or spreading powers on insects. This recalls French Patent 640,736 of 1927, granted to British Dyestuffs, J. Baddiley and E. Chapman, who used sulphonated petroleum products condensed with isopropyl alcohol; the connection with de Ong's work (above) may also be noted.

It may be noted at this point that the inclusion in a liquid of resinous extracts from petroleum, unsaturated and aromatic hydrocarbons, including cracked fractions and solvent-extracted aromatics from kerosine, has been found to improve the toxicity and spreading effect of films.

The ethanolamine soaps have come into prominence lately as emulsifiers (See R. B. Trusler, *Ind. Eng. Chem.*, 1929, **21**, 685, also Patent application 27,425 of 1927, by I. G. Farb. A.G.) and G. L. Hockenyos[76] in a preliminary report has suggested the advantages of using triethanolamine oleate as a desirable stabiliser of oil spray emulsions.

This author also refers to important work by Britton[77], also by Griffin, Richardson and Burdette[78] on oil emulsions for sprays.

E. N. Cory[79] uses pine oil solutions of the popular para dichlor benzene emulsified with soap against the peach borer and states that the higher fractions of pine oils seem to be the more toxic.

Many other substances are mentioned in the literature including:—

Derris Root Extracts (S. Takei, U.S.P. 1,724,626; *Chem. Abstr.*, 1929, **23**, 4766).
Delphinium Extracts (W. M. Davidson, *J. Econ. Entomol.*, 1929, **22**, 226).
Carbon Disulphide (E. N. Cory, *Chem. Abstr.*, 1929, **23**, 4293).

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[74] Fr.P. 654,416/654,419.
[75] *Ind. Eng. Chem.*, 1929, **21**, 542.
[76] *Ind. Eng. Chem.*, 1929, **21**, 647.
[77] *J. Econ. Entomol.*, 1928, **21**, 419.
[78] *J. Agr. Res.*, 1928, **34**, 737.
[79] *Chem. Abstr.*, 1929, **23**, 4293.

Chloropicrin (A. Piedallu and A. Balachowsky, *Chem. Abstr.*, 1929, **23**, 4294) (I. Levy, Fr.P. 658,491).
Cracked Products (Bataafsche P. M., Fr.P. 658,639).
Alkyl Phthalates (W. Moore and H. E. Buc, U.S.P. 1,727,305).
Thiocyanates (O. Y. Imray and I. G. Farb. A.-G., E.P. 317,525).

Naphthenic Acids.

The literature shows comparatively little advance in this subject.

T. Kuwata[80] has continued work on the constitution of naphthenic derivatives from Japanese petroleums by reduction and oxidation. From undeca-, dodeca- and octanaphthenic acids, various five and six carbon ring naphthenes or their derivatives have been obtained. Methods include reduction through alcohols and iodides to hydrocarbons with the aid of platinum black at 300°C., also oxidation by lead peroxide.

G. Komppa, O. Routala and J. M. Kanerva [81] have examined naphthenic acids present in Baku petroleum and, while attempting to reduce or convert them into naphthenes similar to those known to occur in this petroleum, have prepared and determined the constants of a number of derivatives.

V. N. Ipatiev and A. D. Petrov[82] have prepared acids and hydrocarbons from naphthenic acids by means of hydrolysis and destructive hydrogenation, while the I.G. Farbenind. A.G.[83] has used naphthenic soaps of ethanolamines as emulsifying agents.

S. Pilat and E. Davidson[84] found that certain naphthene sulphonic acids formed in the acid refining of oils and having formulæ approximating to $C_{24}H_{33}SO_3H$ did not lose the sulphonic group even on prolonged boiling with alcoholic alkali.

Several references have dealt with the separation of naphthenic acids from residues, crude oils or fractions by means of alkali or water extraction, distillation of volatile substances from salts so produced and subsequent liberation of the acids from the salts.

Subject to variations, the following are on such lines :—

Y. Tanaka and T. Kuwata (*J. Fac. Eng. Tokyo*, 1928, **17**, 293 ; *Brit. Chem. Abstr.*, B, 1929, 117).
C. Arnold and Humble Oil & Refining Co. E.P. 304926.
N. V. Bataafsche Petr. Maatschappij. E.P. 302346.
S. P. Coleman. U.S.P. 1720821.
V. Shiperovich and V. L. Gurevich (*Chem. Abstr.*, 1929, **23**, 1508),

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[80] *J. Fac. Eng. Tokyo*, 1928, **17**, 305, 311 ; *J. Inst. Petr. Techn.*, 1929, **15**, 56A.

[81] *Ber.*, 1929, **62**-B, 1562 ; *Chem. Abstr.*, 1929, **23**, 4935.

[82] *Chem. Abstr.*, 1929, **23**, 4453.

[83] E.P. Application, 27,425 of 1927.

[84] *Brit. Chem. Abstr.*, B, 1929, 768.

Oxidation and Oxidised Products.

The vast amount of work published on the oxidation, flame and explosion characteristics of the lower hydrocarbons in connection with their behaviour as fuels, together with other work on syntheses from the lower hydrocarbons (often through the intermediate stages of oxides of carbon) do not come under the purview of this section.

The commercial production of methanol has received attention in the general work classified below (see also E.P. 272,864, *J. Inst. Petr. Techn.*, 1929, **15**, 240A), but comparatively few papers have indicated material progress towards applications of processes on a commercial scale for the utilisation of oxidation products from petroleum.

G. Egloff and R. E. Schaad[85] indicate potentialities in the production of alcohols, aldehydes, acids, resins, etc., from the vast quantities of natural gas and other available material. A suggested programme of organised study deals with mechanism, equilibrium, catalytic oxidation, chemical and bacterial oxidation with problems of possible commercial value.

The same authors with C. D. Lowry[86] discuss the first item of the above, reviewing the history and relative merits of the hydroxylation and peroxide theories.

W. P. Bitler and J. H. James[87] have given details of the working of the vapour phase oxidation plant at Nyack, dealing in four units with a throughput of 4,000 gallons a day. With a good catalyst, oxidation begins at 225°C. "Aldehol" denaturant is made from the oxidation products and there is sufficient formaldehyde to enable recovery from water as hexamethylenetetramine. Nitrocellulose solvents, plasticisers, softeners and other products can be manufactured. (A note on this appeared in the 1927 naphthology progress reports.)

C. Ellis[88] has patented a vapour phase oxidation method with air over catalysts such as copper gauze, aluminium or vanadium just below red heat for the production of aliphatic acids.

W. E. Messer[89] has prepared alcohols and acids from synthetic high boiling olefines.

C. H. Bibb and H. J. Lucas[90] oxidise gaseous and liquid hydrocarbons to alcohols, phenols and such like by air in the presence

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[85] *Oil and Gas J.*, March 14, 1929, 156.
[86] *Ind. Eng. Chem.*, 1929, **21**, 785.
[87] *Chem. Met. Eng.*, 1928, **35**, 156; *J. Inst. Petr. Techn.*, 1928, **14**, 638.
[88] U.S.P. 1,697,262/7.
[89] *Oil and Gas J.*, April 11, 1929, 130.
[90] *Ind. Eng. Chem.*, 1929, **21**, 633.

of nitric acid or oxides of nitrogen. Speed is an important function, *e.g.*, the heating time for the production of formaldehyde from natural gas is under half a second at 700-750°C.

Oxides of nitrogen, ozone, chlorine or other agents are used under the influence of a high potential electrical discharge in the presence of a catalyst to assist the vapour phase oxidation of gas oil or kerosine by G. Egloff and J. C. Morrell.[91]

Oxidation patents also include those granted to the Selden Company[92], who use base exchange substances, and to J. H. James[87], [93], who obtains dibasic acids.

S. Pilat and J. Winkler[94] find that the optimum temperature of formation of olefinic gases convertible into alcohols from the cracking of gas oil in the vapour phase is 600-670°C.; thus, the temperature most favourable for substances from which good alcohol yields can be obtained is also that for the yield of gasoline, a result which is opposed to that found in previous literature of the subject.

Unpublished work carried out recently has shown the formation of formic acid during the distillation of petroleum in refineries; condensed water accompanying worm end products may hold appreciable quantities, while air blowing of fractions right up to lubricants has produced similar results.

In this connection also, F. W. Hall and R. de M. Taveau,[95] before an A.P.I. meeting at Chicago (December, 1928), discussed the formation of fatty acids during cracking and distillation. Large concentrations of acetic and higher acids were obtained by distilling an evaporated water co-distillate with concentrated sulphuric acid; probably formic acid had been produced, but destroyed, by this treatment.

V. Voorhees, in the discussion, mentioned that he had noticed the formation of formic and oxalic acids when blowing gasoline at water bath temperatures.

The same subject is discussed in F. Hebler's[29] review of oxidation processes; he quotes previous work of Beryman, Frank, Schreyer, Kragen and others, who had obtained lower and higher fatty and naphthenic acids by blowing various petroleum fractions. The existence of free fatty acids in certain crude petroleums is noted by Y. Tanaka and T. Kuwata.[96]

Some of the numerous oxidation patents taken out by the I.G.

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[91] U.S.P. 1,710,155.
[92] Fr.P. 649,292; see *Chem. Abstr.*, 1929, **23**, 2985.
[93] U.S.P. 1,721,958/9.
[94] *Chem. Abstr.*, 1929, **23**, 4560.
[95] *Oil and Gas J.*, December 6, 1928, 110.
[96] See *J. Inst. Petr. Techn.*, 1929, **15**, 55A.

Farbenindustrie A.G. have been mentioned under wax; another is E.P. 301,000 for the production of alcohols by hydrolysis from the unsaturated products of destructive hydrogenation of oils, coals, etc.

The improvement of lubricating oil bottoms to "Viscozins" of high viscosity by air and steam blowing at 302°C. is mentioned by P. Montag,[97] while A. D. Demchenko and C. N. Obryadchikov[98] oxidise viscous bottoms to pitch in much the usual manner for such operations. Other references to pitch blowing occur in the next sub-section.

An interesting application of oxidation during cracking for the prevention of coke formation, which is avoided by the use of well distributed, finely divided air bubbles, has been patented by E. E. Reid,[99] while another method worthy of mention is that of W. W. Gavy,[100] who uses nascent oxygen obtained through the agency of SO_3 produced and decomposed *in situ*.

This patent claims the oxidation of sulphurous and deleterious products without undue waste in the form of sludge by what is practically a combination of a sulphuric acid contact plant in the controlled oxidation sphere, where SO_2 and oxygen are made to combine in the presence of platinised asbestos, the SO_3 formed afterwards decomposing and effecting the oxidations.

S. Mizushima and T. Yamada[100a] have found that sulphur and selenium act as anti-oxygenation and anti-reversion agents when added to waxes and transformer oils.

Asphalt, Pitch and Bituminous Emulsions.

The dominant note in most of the general papers on applications of the above to road construction is the vital need for further co-operation between the chemist and engineer. There is now evidence of a definite advance towards a fuller understanding of scientific applications in this sphere.

General Papers.—An account of the occurrence and refining me thodsof the native asphalts of Trinidad and Bermudez (Venezuela) is given by J. S. Miller,[101] who, after giving data on composition, shows how the substances are refined from water and H_2S. Owing to its high mineral content, refined Trinidad asphalt has a penetration of 2, against 20 for Bermudez material, and these native products are fluxed with petroleum asphalts for commercial use.

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[97] *Chem. Abstr.*, 1929, **23**, 4564.
[98] *Chem. Abstr.*, 1929, **23**, 4335.
[99] U.S.P. 1,663,523; *J. Inst. Petr. Techn.*, 1928, **14**, 189A.
[100] U.S.P. 1,667,440; *J. Inst. Petr. Techn.*, 1928, **14**, 255A.
[100a] *Brit. Chem. Abstr.*, B, 1929, 931.
[101] *J.S.C.I.*, 1929, **48**, 782.

(See also *Petr. Z.*, 1929, **25**, 415, and E.P. 279,026.) It is pointed out that there is need of further knowledge on "cohesiveness" and "adhesiveness," as representing two of the most important properties of asphalts for road building and other surfacing needs.

As a rule, the more adhesive an asphalt the greater is its change in softness with rise of temperature.

H. M. Langton[102] has described the occurrence of Gilsonite in Colorado and Utah, with a few remarks also on the classification of native bitumens.

F. Wilhelmi[103] has given a detailed résumé of road-making emulsions, with particular reference to the specifications of German products.

The functions upon which successful use depends are purity of the bitumen, stability of the emulsion, fineness and non-tendency to re-emulsification of the bitumen liberated *in situ*.

There are many differences between various kinds of coal tar and asphalt pitches, some of which require alkali soaps, caseinates, sulphonates, etc., while the stabilising effect of such colloids as glycerin, dextrin, proteins and such like are noted.

Among the materials used in Germany, a special emulsion known as Teeramuls consists of 90 to 95 per cent. tar to which asphalt may be added, together with not more than 2 per cent. of water and 5—10 per cent. of emulsifier; this jellies in the cold and must be used hot.

P. Hubbard[104] has discussed the chemistry of bituminous highway construction, with reference to pitches and road oils.

The effectivity of a petroleum asphalt in road-making depends on its origin and refining treatment; for example, the effect of prolonged heating in shell stills is bad, though a judicious use of oxidation by air blowing may be the best finishing treatment.

There may be great differences between blown asphalts from poorly stripped and well-stripped residues, even if taken to similar penetration values.

An important study is that of the adsorptive character of sand or other aggregate, and it seems that the best results are connected with high adsorptive values. The paper concludes with a study of the application of residual or cut-back pitch-oil mixtures.

H. Suida and A. Schmölzer[105] have also studied the adsorptive properties of aggregate fillers such as limestone, granite, quartz and brick in relation to the plastic and elastic properties of native

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[102] *Industr. Chem.*, 1929, **5**, 324.
[103] *Erdol u. Teer*, 1929, 87; *J. Inst. Petr. Techn.*, 1929, **15**, 80A.
[104] *J.S.C.I.*, 1929, **48**, 1087.
[105] *Petr. Z.*, 1929, **25**, 251; *J. Inst. Petr. Techn.*, 1929, **15**, 82A.

asphalts, coal tar and petroleum pitches. The softening property of limestone as a filler to coal tar pitch is due to the reactive acid constituents in that pitch, which are absent from petroleum or native asphalts. Asbestos also differs from other fillers owing to its fibrous structure, which is favourable to elasticity.

A. W. Dow[106] also deals with the adsorption of aggregate on asphalt paving mixes, and states that the higher adsorptive substances are best; the property may be tested by means of carbon disulphide solutions.

E. S. Ross[107] has compiled information on the use of asphalt emulsions. Softer grades of asphalt are used for emulsions, such as those with a penetration at 77° F. of 35 to 200, softening point 110–160° F. The colloidal state of the emulsion is determined by interfacial tensions of the particles and the medium, viscosities and hydrogen-ion concentrations as fundamental factors.

In bitumen-in-water emulsions the globules carry a negative charge; hence alkali increases the difference in potential by adsorption of OH ions on the suface of the protective colloid.

If clays such as bentonite are added, care must be taken to avoid those more easily wetted by oil than water; otherwise the emulsion may tend to invert. A suitable emulsion must withstand destruction by freezing, must penetrate, break down easily through physical change *in situ* without the necessity of drying out, and must not tend to re-emulsify when dry.

W. J. A. Butterfield[108] discusses the changed conditions of road use and the use of various materials. Petroleum bitumens differ from coal tars in having no "drying" or "setting" constituents as do the latter. Desirable and undesirable constituents are noted with reference to dangers to fish life in near-by streams.

W. E. Cone[109] has also criticised the use of road tars in Great Britain with reasons for some failures.

P. E. Spielmann[110] has given an account of the findings of the Paris meeting of the International Committee on nomenclature of materials and processes used in the construction of roads and the standardisation of testing methods for tars, bitumens and asphalts. The Committee agreed on various definitions and tests, but drew attention to the need for further work on the quality of stabilisers and on tests for plasticity, adhesion, cohesion, oxidation and atmospheric resistance. The definitions adopted were those which had been usually understood in Great Britain, except that the

[106] *Chem. Abstr.*, 1928, **22**, 2253.
[107] *J.S.C.I.*, 1929, **48**, 112T.
[108] *J.S.C.I.*, 1928, **47**, 293T.
[109] *J.S.C.I.*, 1928, **47**, 1048.
[110] *J. Inst. Petr. Techn.*, 1928, **14**, 774.

existing American definition was adopted to describe bitumen, the only practical limitation of which is solubility in carbon disulphide.

W. Bain[111] has discussed the wetting of asphalts and the importance of surface tension effects on the stability of asphalt road emulsions before and after use in conjunction with the adsorptive power of aggregates. Surface tension is found to be lowest with oxidised asphalt, higher with petroleum residues, and highest for coal tar, these differences probably accounting for many troubles experienced when changing from one to the other. Natural asphalts have held their own against the purer artificial products because of their perfect wetting power on aggregates.

F. J. Nellensteyn[112], in a paper on relation of the micelle to the medium in asphalt, has touched upon some of the same points, and gives the same order of surface tensions for blown asphalt, non-blown petroleum residues and coal tar.

This paper also views the precipitation of asphalt from petroleum mixtures or solutions in various lights, according to the solvents and precipitants used.

The same author with J. P. Kuipers[113] has also studied the ultramicroscopy of asphalts in solution, with attention to the effects produced by adding finely divided materials.

Several papers on the surface treatment of roads, effect of Aggregates on Stability of Bituminous Mixtures, Latest Developments in Asphalt Paving Practice, and other similar subjects are adequately abstracted in *J. Inst. Petr. Techn.*, 1929, **15**, 321A–326A, from the A.S.T.M. Symposium on Mineral Aggregates (June, 1929) and various journals on Congress reports.

Among numerous other references, brief mention is made of the following :—

L. Kirschbraun.[114] Use of asphalt emulsions in chemical plant, proofing, coating of surfaces, reduction of fire hazards, etc.

G. K. Kropfhammer.[115] The stability of tars and bitumens, wetting of road surfaces by preliminary use of emulsions followed by hot pitch, the use of hexaline and homologues in emulsification.

Von Skopnik.[116] The chemistry of road construction materials, testing and practice.

E. T. Leemans.[117] A review of patents on road materials and coating compositions from 1907 to 1928.

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[111] *J.S.C.I.*, 1928, **47**, 552.

[112] *J. Inst. Petr. Techn.*, **15**, 134.

[113] *Koll Z.*, 1929, **47**, 155 ; *Chem. Abstr.*, 1929, **23**, 3567.

[114] *Chem. Met. Eng.*, 1929, **36**, 477.

[115] *Erdol u. Teer*, 1928, **29**, 513 ; *J. Inst. Petr. Techn.*, 1928, **14**, 329A.

[116] *Chem. Z.*, May 19, 1928.

[117] *Chem. Weekblad*, 1929, **26**, 2.

G. B. Chilvers and O. N. Edwards.[118] Cold asphalt emulsions.
R. Grun.[119] Asphalt coatings on concrete.
H. Mallison.[120] Chemistry of road tar with photomicrographs.
W. Von Piotrowski.[121] Use of road oils as dust preventives with notes on their penetrating power.
I. Pedersen.[122] Use of asphalt emulsions at Copenhagen.
S. Uchida.[123] The influence of asphalt emulsions on the time of setting and strength of cement, delay in setting and loss of strength caused by using asphalt emulsions instead of water.
C. E. Kern.[123a] The increasing importance of road oil manufacture; and research programme of U.S. Bureau of Roads.
W. G. Adam and J. S. Sach.[123b] Conditions affecting the formation of fine carbon in coal tars and pitches with microphotos and notes on a pyridine solubility test for overburning.
In addition, most of the larger petroleum and asphalt companies or groups of companies have issued useful trade pamphlets on the use of bitumens in the form of pitches, oils and emulsions, so that much of their practical experience, which has developed considerably during the period under review, is now available in this form. The catalogues of many instrument makers also provide summarised information on the testing of these products.

Manufacture of Asphalt. G. W. Cupit[124] published a well detailed essay on the nature, manufacture, blowing and testing of asphalt; a somewhat less detailed summary was also given by D. F. Gerstenberger.[125]

W. W. Peters[126] gave methods and specifications for the manufacture of roofing asphalts, blown and unblown, from selected crude oils with a description of accelerated tests in which weaknesses could be demonstrated.

N. Williams[127] gave a detailed account of the methods in use at a New Orleans plant for the production of asphalts to meet various specifications pointing out the importance of flexibility in temperature control.

A. N. Sakhanov and L. G. Sherdeva[128] reviewed problems and difficulties in the manufacture of asphalts from acid sludges.

Testing Methods.—The paraffin content of asphalts for road making was dealt with by H. Burstin[129], who differentiated between crystalline wax which is harmful, and amorphous material which is more elastic and akin to the asphalt itself and therefore far less

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[118] *Munic. Eng. Sanit. Record*, 1928, 688, 702.
[119] *Chem. Abstr.*, 1928, **22**, 3030.
[120] *Petr. Z.*, 1929, **25**, 299; *Chem. Abstr.*, 1929, **23**, 2563.
[121] *Petr. Z.*, 1929, **25**, 260; *Chem. Abstr.*, 1929, **23**, 2564.
[122] *Chem. Abstr.*, 1929, **23**, 686.
[123] *Chem. Abstr.*, 1929, **23**, 2799.
[123a] *Oil and Gas J.*, November 14, 1929, 154.
[123b] *J.S.C.I.*, 1929, **48**, 337T.
[124] *Refiner*, August, 1928, 81; November, 1928, 76.
[125] *Nat. Petr. News*, February 8, 1928, 81.
[126] *Oil and Gas J.*, March 7, 1929, 113.
[127] *Oil and Gas J.*, August 16, 1928, 153.
[128] *Chem. Abstr.*, 1928, **22**, 2461.
[129] *J. Inst. Petr. Techn.*, 1929, **15**, 137A.

harmful. Three methods of determination were discussed, those of a cracking method, the method of Schwarz and that of Marcusson.

H. Bösemberg[130] studied various standard methods of testing bitumens for the usual properties; A. Spilker[131] determined the softening point with the aid of a rod of type metal (80Pb/20Sb) in order to avoid the possibility, suggested by Mallison, of mercury poisoning in the Kraemer-Sarnov method, at the same time retaining comparative results. A contribution to this discussion had been given by D. Holde[132] but G. M. Green[133] advocated a softening point method depending on the separation of the melting asphalt from a pin point upwards through a heavier and non-reacting liquid.

Specific gravity determination methods were dealt with by S. E. Berkenblit[134] also by H. R. Berry, E. H. Smith and F. R. Lang[135].

Patent Literature.—A large number of patents has appeared of which the following may be noted :—

(a) *Use of Bituminous Emulsions.*

E.P. 279,026. Soc. An. la Trinidad. Trinidad asphalts with colloidal mineral substance fluxed correctly with a residual petroleum oil, incorporated with 3–12 per cent. anhydrous soap containing commercial olein and emulsified.

E.P. 291,393. Bataafsche Petr. Maatschappij. Use of aromatic sulphonic acids.

E.P. 292,251. Asphalt Cold Mix Co. Use of oleic acid, silicates, borates, etc.

E.P. 298,842. Chem. Fabr. Buckau. Use of acids or oxyacids of boron up to 2 per cent.

E.P. 300,414, 300,415, 300,821, 305,716, 308,051. J. A. Montgomerie. Use of sodium peroxide, cresylates, trisodium phosphate, vegetable or animal pitch, with sodium laurate as a stabiliser.

E.P. 301,891. W. H. Schmitz. Wetting stone aggregate with oil, then following with emulsion.

E.P. 303,281. I. G. Farb. Ind. A.-G. Use of sulphonated oxidation products of petroleum.

E.P. 305,742. E. C. R. Marks. Use of resins and milk of starch in preparation of emulsifiers.

E.P. 308,389. T. M. Hickman. Use of alkali caseinate with rosin and an ingredient to prevent fermentation.

E.P. 315,495. G. C. Hurrel. Use of clays as stabilisers.

E.P. 317,496, F.P. 652,114. I. G. Farb. A.-G. Use of humic acid.

F.P. 648,917. V. Wintsch. Incorporation of latex in bituminous emulsions for roads.

F.P. 655,355. Flintkote Co. Regulation of pH value and use of clays.

U.S.P. 1,679,475, Can.P. 279,241. L. Kirschbraun. Use of bentonite.

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[130] *Petr. Z.*, 1929, **25**, 1127, 1275; *J. Inst. Petr. Techn.*, 1929, **15**, 268A, 376A.
[131] *Petr. Z.*, 1929, **25**, 835; *Z. Angew. Chem.*, 1929, **42**, 263.
[132] *Petr. Z.*, 1929, **25**, 411.
[133] *J.S.C.I.*, 1929, **48**, 114T.
[134] *Ind. Eng. Chem. Anal.*, 1929, **1**, 39.
[135] *Ind. Eng. Chem. Anal.*, 1929, **1**, 164.

U.S.P. 1,691,766. L. Kirschbraun and H. L. Levin. On the importance of H-ion concn. control.

U.S.P. 1,699,536/7. Jones, Loomis and Banks. Addition of asphalt-soap-gasoline paste to blown asphalt.

U.S.P. 1,706,590. H. Plauson. Emulsification of bitumen with previously emulsified material.

U.S.P. 1,707,809. L. Kirschbraun. Bituminous emulsions for paper manufacture, use of fibrous materials, clays and salts.

U.S.P. 1,714,982. A. L. Halvorsen and P. M. Travis. Use of sodium phosphate.

U.S.P. 1,725,648. L. Kirschbraun. Use of alcohols and deflocculating electrolytes.

U.S.P. 1,726,708. L. Kirschbraun. Use of emulsions in the surgery.

(*b*) *Manufacturing Methods for Oxidised Asphalts.*

U.S.P. 1,667,731. C. Sautermeister and F. Wilhelmi. Preparation from acid sludge by H_2S or sulphur.

U.S.P. 1,673,533. E. A. Rudigier. Details of tanks, blowers, etc., for manufacturing air blown asphalts.

U.S.P. 1,715,069. L. Kirschbraun. Manufacture of oxidised asphalt by passage with air through a temperature graded conduit and delivery to a tank in which agitation is maintained.

U.S.P. 1,718,679. J. C. Black and W. D. Rial. Method of air blowing pitches.

U.S.P. 1,735,503. L. Kirschbraun. Manufacture of Asphalt.

(*c*) *Coating Compositions.*

E.P. 289,737. G. A. Wellings and E. Johnstone. By adding sulphur in solution or suspension in a solvent to a solution of bitumen.

E.P. 301,544. Colas Products, etc. Application of bituminous emulsions to mineral aggregates, cork, asbestos, etc., in the presence of a strong electrolyte.

U.S.P. 1,678,980. J. B. W. Gardner. Application of bituminous emulsions to wood, metal or masonry and while wet, coating with dry cement which sets with the water liberated from the emulsion.

U.S.P. 1,691,765. L. Kirschbraun. Asphalt emulsion paints.

U.S.P. 1,705,015. H. H. Irving. Roofing material from asphalt and tar pitches.

F.P. 658,095. McRae Paint Products. Use of Gilsonite with petroleum asphalts and fractions, also with pigments if desired.

(*d*) *Miscellaneous Paving Aggregates, Road Oils, Reinforced Compositions.*

There is very little novelty in the numerous patents granted. The use of rubber, mineral aggregates, addition of small quantities of emulsion to pitches to prevent premature hardening, reinforcement of bituminous compositions with cement or on metal frames, paving compositions and methods of application form the subjects of the following and others :—

E.P. 290,725 (Brown), 290,886 (Fuery), 292,871 (Hines), 293,830 (Berger), 299,208 (Stein), 300,828 (Capmany), 304,114 (N. V. de K. S. K. Gouda).

U.S.P. 1,662,377 (Downard), 1,681,353 (Levy), 1,717,445 (Flood).

F.P. 655,858 (Stadt. Dresden).

Miscellaneous.

References selected from the increasing literature on miscellaneous products are tabulated below with indications regarding their contents :—

Acid Sludge.

Disposal by burning. Faught and Staley. *Refiner*, February, 1928, 73.

Review on disposal. Rather. *Refiner*, April, 1928, 88; *Oil and Gas J.*, March 1, 1928, 102.

Separation and burning. Osborn and Davis. *Refiner*, December, 1928, 109; *Oil and Gas J.*, December 6, 1928, 140; *Nat. Petr. News*, December 5, 1928, 106.

Fluxing with fuel oil. Barbour. *Oil and Gas J.*, December 6, 1928, 242.

Disposal by burning. Kiehl. *Oil and Gas J.*, December 13, 1928, 139.

Disposal. Ziegenhain. *Oil and Gas J.*, January 24, 1929, 132.

Use as binder for briquettes. Johnston and Farrell. *Oil and Gas J.*, April 4, 1929, 188.

Determination of free acid. Bacon. *Ind. Eng. Chem. Anal.*, 1929, **1**, 89.

Disposal. Truesdell. *Nat. Petr. News*, June 26, 1929, 80.

See also *J. Inst. Petr. Techn.*, 1928, **14**, 5A, 163A, 268A; *Chem. Abstr.*, 1929, **23**, 4810, 4812, and Section on Asphalts.

Alcohols. See oxidation products (above), also General (below), also note on production of Amyl Alcohols from Pentane. Ayres. *Ind. Eng. Chem.*, 1929, **21**, 899.

Aluminium Chloride.

Manufacture by briquetting with coal or coke and chlorination. McAfee *Ind. Eng. Chem.*, 1929, **21**, 670.

Amyl Acetate.

From natural gas pentane by chlorination and esterification. Dobryanskii and Gurevich. *Chem. Abstr.*, 1929, **23**, 4811.

Use to prevent gumminess in lubricants. Sanders. E.P. 297,201 (*J. Inst. Petr. Techn.*, 1928, **14**, 328A).

Asphalt Varnish.

By blowing acid sludge with turpentine and wood tar. Karskii and Kapatzinskii. *Chem. Abstr.*, 1929, **23**, 4812.

Bentonite.

Properties and uses. Woodman and Taylor. *J.S.C.I.*, 1929, **48**, 121T.

Butadiene and Diolefines.

Preparation of butadiene. Birch. *Ind. Eng. Chem.*, 1928, **20**, 474.

Production of olefines and diolefines. J. T. Johnson. E.P. 303,998.

Catalytic preparation of butadiene and diolefines with steam, etc. J. Y. Johnson, I. G. Farbe A.-G. E.P. 269,593, 292,103, 303,323, 307,945, 313,756, 315,595, 315,895, 316,422, 317,359, 317,500, 319,025.

Manufacture. Nash, Bowen and Elvins. E.P. 291,867.

Carbon Black and Lamp Black.

Study of volatile constituents. C. R. Johnson. *Ind. Eng. Chem.*, 1928, **20**, 904.

Smokeless carbon by partial combustion of natural gas. General Atlas Chemical Co. *Oil and Gas J.*, December 6, 1928, 237.

General article on lamp black. Grove Palmer. *J.S.C.I.*, 1929, **48**, 60.

Abstract of methods of preparation from acetylene. Anon. *Industr. Chem.*, 1929, **5**, 121.

Preparation from residue gas of gasoline plants. Cabot. *Oil and Gas J.*, May 2, 1929, 78.

From natural gas. Yunker. *Oil and Gas J.*, May 9, 1929, 111.

By combustion of liquid hydrocarbons on trays. Major. E.P. 303,632.

Use in rubber industry. Carson and Sebrell. *Ind. Eng. Chem.*, 1929, **21**, 911.

Determination of moisture content. Carson. *Ind. Eng. Chem. Anal.*, 1929, **1**, 225.

Grinding with tar to produce a coherent mass. Comp. Lorraine de Charbons pours l'Electricite. E.P. 300,610.

Lecture on processes and uses. Langton. *Oil Col. Trades J.*, 1919, **76**, 1273.

Adhesion tension against water and benzene. Bartell and Smith. *Ind. Eng. Chem.*, 1929, **21**, 1102.

Role of Oxygen in Carbon Black. C. R. Johnson. *Ind. Eng. Chem.*, 1929, **21**, 1288.

Manufacture of CO. J. Y. Johnson and I.G. Farb. A.-G. E.P. 319,140.

Comparison of Acetylene Black with Gas and Lamp Black. Dawson and Hartshorn. *J. Inst. Petr. Techn.*, 1929, **15**, 369A.

Cement or other Anti-Corrosive Coatings for Metal Plant or Pipes.

Pipe-line protection. Hagen. *Oil and Gas J.*, May 10, 1928, 114.

Application of pipe-line coatings. Anon. *Oil and Gas J.*, June 7, 1928, 39. Finley. *Oil and Gas J.*, October 25, 1928, 126.

Pipe-line and soil corrosion. Logan. *Oil and Gas J.*, August 30, 1928, T 200.

Bituminous coatings, etc. *Oil and Gas J.*, May 2, 1929, 33.

Pipe-line protection. Van A. Mills. *Oil and Gas J.*, August 30, 1928, T 48.

Use and behaviour of pipe-line coatings. Scott. *Oil and Gas J.*, December 6, 1928, 127. *Petr. World*, 1929, **26**, 49.

Cement-silicate-asbestos protection of pressure plant. Morrell Faraghor and Mekler. *Oil and Gas J.*, December 6, 1928, 55.

Pipe-line coatings. Sub-Committee Report. *Refiner*, January, 1929, 117.

Protection of underground pipes in California. Slater. *Ind. Eng. Chem.*, 1929, **21**, 19.

Bituminous coatings. Messrs. Stewart & Lloyds. E.P. 300,933.

Protective coatings including internal coating of pipes. Speller. *Ind. Eng. Chem.*, 1929, **21**, 508.

Pipe-line coatings. Bignell. *Oil and Gas J.*, August 29, 1929, 35.

(See also Asphalt and Bituminous Emulsion sub-section.)

Chlorination and Chlorinated Products.

Manufacture. Vivas. E.P. 286,726 (*J. Inst. Petr. Techn.*, 1929, **15**, 143A).

Chlorination and catalysis. Int. Fireproof Products. F.P. 650,738 (*Chem. Abstr.*, 1929, **23**, 3341).

Production through hydrogenation. I. G. Farb A.-G. E.P. 301,009 (*Chem. Abstr.*, 1929, **23**, 4050).

Preparation of alkylene chlorhydrins. Th. Goldschmidt A.-G. E.P. 303,764 (*J. Inst. Petr. Techn.*, 1929, **15**, 244A).

Cleaning Spirits (Stoddard Solvent).

Specifications for cleaning specialties. Francis. *Oil and Gas J.*, February, 1928, 260.

Specification. Anon. *J. Inst. Petr. Techn.*, 1928, **14**, 24A.

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Purification. Hatfield. *J. Inst. Petr. Techn.*, 1929, **15**, 83A.

Purification by soda solution followed by solid $CaCl_2$. Boon. U.S.P., 1,711,829.

Purification by vacuum, distillation and centrifuging. MacLeod. U.S.P. 1,720,604 (*J. Inst. Petr. Techn.*, 1929, **15**, 266A).

Cleaning Compound for Cylinders of I.C. Engines.

Mixtures of naphthalene and sod. carbonate. Mellors. E.P. 304,923.

Dyes.

Use of dyes for analysis of hydrocarbon classes in mixtures. Formanek. *Chem. Zeit.*, 1928, **52**, 325; *Refiner*, September, 1929, 139.

Electrical Applications in the Petroleum Industry (see also Emulsions).

Effect of cathode rays. Schoepfle. *Ind. Eng. Chem.*, 1929, **21**, 529.

Effect of spark discharge on methane. Stanley and Nash. *J.S.C.I.*, 1929, **48**, 238T.

Effect of semi-corona discharge. Lind and Glockler. *J. Amer. Chem. Soc.*, 1928, **50**, 1767; 1929, **51**, 2811.

Electrification of oil fields. Toplis. *J. Inst. Petr. Techn.*, 1929, **15**, 591; *Petr. Times.*, 1929, **22**, 721.

Polymerisation of lower olefines to valuable unsaturated products by high temperature electrical discharges. Johnson and I. G. Farb. A.-G. E.P. 315,249 (*J. Inst. Petr. Techn.*, 1929, **15**, 241A).

Action of electric arc on liquid hydrocarbons. Mercier. E.P. 325,336. 316,352.

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Chemical reactions in gases by electrical discharges. Hull and I.C.I. E. P. 317,920.

Synthesis by B-rays. Potts. E.P. 309,002.

Production of lubricating oils from cracking gases. Epner. E.P. 294,099 (*J. Inst. Petr. Techn.*, 1929, **15**, 260A).

Production of lubricating oils of high viscosity. I. G. Farb. A.-G. E.P. 303,761, 305,553, 303,776.

Properties and viscometry of "Voltol" oils. Ostwald and Fohre. *Koll. Z.*, 1928, **45**, 266; *Brit. Chem. Abstr. B*, 1929, 700; *J. Inst. Petr. Techn.*, 1929, **15**, 69A-71A.

Voltol oil manufacture. Deutsche Voltolwerke. *Chem. Zeit.*, August 15, 1928.

Abstract on Voltols. Kauffman. *Refiner*, July, 1929, 122.

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Theory and practice of treatment. Catlin. *Refiner*, June, 1928, 57.

Electrical dehydration in Argentina. Wyant. *Nat. Petr. News*, October 17, 1928, 119.

Use of filter aids. Elsenbast and Horine. *Oil and Gas J.*, January 10, 1929, 140.

Simple salt water method for breaking. Van A. Mills. *Oil and Gas J.*, April 25, 1929, 45.

Oil field emulsions, a comprehensive study. Uren. *Nat. Petr. News*, 1929, March 27, April 17, May 15, July 3.

Industrial applications. Meunier. *J. Inst. Petr. Techn.*, 1929, **15**, 116A.

Breaking methods, a comprehensive discussion. Lottermoser and Calantar. *J. Inst. Petr. Techn.*, 1929, **15**, 191A.

An electric dehydration method. Cage. *Petr. Times.*, 1929, **22**, 394.

Emulsifiers from sulphonated higher aromatics. Bataafsche Pet. Maat. E.P. 291,393; Fr.P. 655,044.

Separation of oils from solids. I. G. Farb. A.-G. E.P. 314,405.

Soaps from sulphonated sludge. Cushman and Doell. U.S.P. 1,718,335.

Breaking by use of chemicals. Bright. *Oil and Gas J.*, June 20, 1929, 40.

Various U.S. patents on breaking. Barnickel. *J. Inst. Petr. Techn.*, 1928, **14**, 92A.

Cracking of emulsions. Egloff. U.S.P. 1,670,107.

Cracking of emulsions. Egloff and Benner. U.S.P. 1,703,103, 1,710,154.

Breaking by other than chemical means. Kuczynski. *Petr. Z.*, 1928, **24**. 398.

Breaking by use of sludge acids. Burgess. U.S.P. 1,668,941; Pester, U.S.P. 1,669,379; Standard Oil Dev. Co. Fr.P. 652,206.

Oil field emulsion, treating. Gardner. U.S.P. 1,668,766.

Breaking by means of powdered granite or slate. Robinson and Parkes. U.S.P. 1,687,314.

Ethanolamine soaps as emulsifiers. Trusler. *Ind. Eng. Chem.*, 1929, **21**, 685.

Mechanical heating-flow method for treating... Moscicki. U.S.P. 1,710,374.

Ethylene Glycol as Antifreezer, etc.

From Canadian natural gas. *Oil and Gas J.*, February 14, 1929, 130.

Note in paper on natural and "cracking" gas. Stanley. *J. Inst. Petr. Techn.*, 1929, **15**, 516.

Use for removal of sulphonated products in refining oils. Moran and Vacuum Oil Co. U.S.P. 1,659,782.

Use of glycol esters as cellosolve in nitrocellulose lacquers. Reid and Hofmann. *Ind. Eng. Chem.*, 1928, **20**, 497.

Grease Manufacture.

General articles by Matthews (*Oil and Gas J.*, March 7, 1928, 148), Faught and Staley (*Refiner*, April, 1928, 72), Kauffman (*Refiner*, July, 1928, 86), Klemgard (*Refiner*, March, 1929, 68), Kauffman (*Refiner*, May, 1929, 93; September, 1929, 106).

Special zinc grease to prevent corrosion. Keenok Gears, Ltd. *Indust. Chem.*, 1929, **5**, 412.

Papers before A.C.S., Minneapolis, September, 1929. Pratt and Weld Koethen. *Nat. Petr. News*, September 18, 1929, 33.

Moisture in calcium greases. Otto. *Oil and Gas J.*, November 14, 1929, 46.

Koethen. *Nat. Petr. News*, October 16, 1929, 69.

Lauge. *Nat. Petr. News*, October 16, 1929, 67.

Green Fluorescence.

Addition of green fluorescence to oils from uncracked pitch or coal tar pitch extracted by oil if necessary. Black, Rial Barratt, McConnell. U.S.P. 1,708,563, 1,708,592, 1,708,602.

Helium (Occurrence in Natural Gas and Monasite Sands, Economics, etc.)

See *Oil and Gas J.*, March 1, 1928, 40; June 14, 1928, 137; September 13, 1928, 130; April 25, 1929, 82; September 12, 1929, 89; September 19, 1929, 90; November 14, 1929, 90; *Petr. Times*, 1929, **22**, 564; *J.S.C.I.*, 1929, **48**, 978, 369T; *Oil and Colour Trades J.*, 1929, **76**, 1110; *Chem. Abstr.*, 1929, **23**, 4654.

Medicinal Paraffin.

For notes on the refining and properties of "White" oil or medicinal paraffin, see *Oil and Gas J.*, September 6, 1928, 134; May 2, 1929, 139; *Refiner*, August, 1929, 77; Burgess, U.S.P. 1,681,895, *J. Inst. Petr. Techn.*, 1928, **14**, 268A.

Nitrogen in Petroleum.

Determination of. Poth, Armstrong, Cogburn and Bailey. *Ind. Eng. Chem.*, 1928, **20**, 53.

Phenol in Petroleum.

Occurrence in Japanese petroleum. Tanaka and Kobayashi. *J. Inst. Petr. Techn.*, 1928, **14**, 146A.

Occurrence in petroleum distillates. Le Roy, Stoup and Snow. *Ind. Eng. Chem.*, 1928, **20**, 359.

Preservative for Timber.

Mixtures of mazout and creosote. Voronov. *Brit. Chem. Abstr. B*, 1929, 768; *Chem. Abstr.*, 1929, **23**, 2802.

Refining Agents (use of special substances other than hypochlorite, SO_2 and silica gel).

Alcohol, anhydrous. Werkenthin. U.S.P. 1,680,352.

Amyl acetate (to prevent gumminess in lubricants). Sanders. E.P. 297,201.

CO_2 liquid. A. G. Fur Kohl. Ind. E.P. 277,946, 285,064.

CO_2 liquid, sol. of lubricating oils in. Quinn. *Ind. Eng. Chem.*, 1928, **20**, 735.

Carbon-clay mixtures. Buttner Werke A.-G. E.P. 295,623.

Ethylene glycol to eliminate sulphonated products. Moran and Vacuum Oil Co. U.S.P. 1,659,782.

Formaldehyde. Nastukoff. E.P. 289,920.

Formation of alcohols. Luther and Pieroh. U.S.P. 1,732,371.

Hydrofluoric acid. Hofmann and Stegemann. E.P. 292,932.

Lead tetræthyl and ethylene bromide to inhibit oxidation of lubricating oil. Callendar King and Mardles. E.P. 295,230.

Persulphuric acid. Axtell. U.S.P. 1,667,425.

Sand (acid treated) (40-60 mesh for lubricating oils, etc.). Maitland. U.S.P. 1,668,920.

Solvents, various. See Processing of wax-oil mixtures.

Substances for inhibiting reversion of kerosine (phenol, urea amines, glycols, etc.). Jobson. E.P. 312,774.

Egloff, Faragher and Morrell. A.P.I. Chicago Meeting, December, 1929, (*Petr. Times*, 1930, **23**, 33.)

Resins and Rubber-like Substances from Petroleum or Derived Products.

From cracked products. Egloff. *Trans. World Power Conf.*, 1928, I, 846.

From cracked products. Hultman. U.S.P. 1,704,194; *J. Inst. Petr. Techn.*, 1929, **15**, 84A.

From oxidised asphalt. Kirschbraun. U.S.P. 1,715,069; *Chem. Abstr.*, 1929, **23**, 3571.

From isoprene and butadiene halides. Baer. E.P. 314,524 (pending).

From butadiene and homologues with latex and hydrogen peroxide at 75° C. Johnson and I. G. Farb. A.-G. E.P. 313,188; *J. Inst. Petr. Techn.*, 1929, **15**, 266A.

Vulcanised products from oil. I.C.I. and Naunton. E.P. 313,252; *J. Inst. Petr. Techn.*, 1929, **15**, 266A.

Synthetic resins from aromatic hydrocarbons by catalysis with zirconium salts, etc. Krishnamurthy. E.P. 298,939; *J. Inst. Petr. Techn.*, 1929, **15**, 58A.

Smoke Clouds.

From lubricating oils with chloroalkyl derivatives and dyes. Savage. U.S.P. 1,716,797.

Special Lubricants (other than Medicinal Paraffin and Voltols).

Cutting oils. Cupit. *Refiner*, June, 1928, 65.

Cutting, water-soluble, leather, textile oils, etc. Hart. *Ind. Eng. Chem.*, 1929, **21**, 85.

Stopcock lubricants for high vacua (rubber-wax-mineral jelly mixtuures). Petzold. *Chem. Abstr.*, 1929, **23**, 966.

Cutting and quenching oils. Hudson. *J. Inst. Petr. Techn.*, 1929, **15**, 68A.

Cutting and soluble oils. Kauffman. *Oil and Gas J.*, June 13, 1929, 46.

Rope wire lubricants (specifications). Meals. *Oil and Gas J.*, June 27, 1929, 40.

Stearic Acid.

Preparation, etc. Francis. *Oil and Gas J.*, October 17, 1929, 85.

Voltol Oils.

See Electrical Applications.

General.

For general papers on research, bye-products, alcohols and commercial possibilities other than those mentioned in the above notes, see :—

Dunstan. Cantor Lectures, 1928.
Brooks. *J. Inst. Petr. Techn.*, 1928, **14**, 738.
Burrell. *Ind. Eng. Chem.*, 1928, **20**, 602.
A.P.I. Programme. *Oil and Gas J.*, March 7, 1929, 120.
Padgett. *Oil and Gas J.*, March 7, 1929, 148.
Klemgard. *Refiner*, June, 1929, 58.
Little. *Oil and Gas J.*, September 19, 1929, 155.
Stanley. *J. Inst. Petr. Techn.*, 1929, **15**, 516.

A feature of the period is also the very great number of patents granted to the I.G. Farbenindustrie A.G., several references to which are included in this section.

Synthetic Fuels.*

By Alfred W. Nash, M.Sc., F.C.S., M.I.Mech.E. (Member).

During the past two years the progress of the research, both academic and technological, was chiefly concerned with two subjects :—

(1) Catalytic berginisation of coal and heavy tarry materials to spirits.

(2) The production of liquid hydrocarbons, from gases such as methane, ethane, propane, etc., directly or the investigation of these gases as potential sources of ethylene and acetylene.

The polymerisation of ethylene to light spirits or heavier oils is of fundamental interest, and may ultimately be shown to constitute a source of synthetic lubricant hydrocarbons. Ethylene and acetylene may also be looked upon as possible sources of alcohol base fuels when such processes reach full technological development.

Again, as was pointed out in the previous report on this subject, the commercial world is still, and should continue to be, well supplied with motor fuel from petroleum, but research upon the subject of synthetic fuels continues with unflagging zeal, especially by the larger chemical organisations.

Catalytic Berginisation of Coal and Heavy Bituminous Substances.

Further details of the process for converting coal, coal tars or other heavy oils largely into hydrocarbons suitable for motor fuels were given by Krauch of the I. G. Farbenindustrie A. G. (2nd Int. Conf. on Bit. Coal, 1928). In discussing the hydrogenation of coal and its distillation products, Krauch points out that this process is an overlapping of cracking, or coking, and hydrogenating process. (It is for the latter reason that the Author prefers the term catalytic berginisation, which would refer to a combined cracking and hydrogenating process, as compared with catalytic hydrogenation where molecules of the reactant are not usually disrupted.) In catalytic berginisation, hydrogen is applied under high pressure,

* Paper received Dec. 30, 1929.

and the control of the temperature of the reaction vessel is important to minimise methane and coke formation. The catalysts used to give flexibility to the process as regards nature of the oil produced and elimination of sulphur and oxygen from combination were, of necessity, to be immune from poisoning, and these, usually difficultly reducible oxides, were developed from the experiments upon the hydrogenation of carbon monoxide and carbon dioxide to methanol and higher alcohols. Thus, this firm claims to have developed catalysts with which coal tars can be converted into stable gasoline with only 10 per cent. of the carbon content lost as gaseous products.

The catalysts are thus immune from the usual poisoning effect of sulphur, and in the pressure hydrogenation of crude oil and coal, such products as gasoline, kerosine, gas-oil and lubricating oils are claimed, in quantities varying with the catalysts employed. No details are given as regards the value of the higher fractions when compared with petroleum fractions; *e.g.*, burning qualities of the kerosine or friction reducing properties and stability of the lubricating oils.

It is stated that coal can be hydrogenated in the form of powder distributed in hydrogen or suspended in a heavy oil, and in the latter case the first stage heavy oil must be separated from the coal ash and unconverted coal particles. At Leuna, where experiments on both the Central German lignite and the low-temperature tar therefrom, and also hard coals from other parts of the world, have been carried out, the conclusion is that all such products are suitable for the manufacture of gasoline.

As regards heavy residues containing sulphur such as those from Panuco crude the sulphur is eliminated very largely as hydrogen sulphide.

The I. G. are stated to be producing approximately 300,000 tons of gasoline per annum by the catalytic berginisation of low-temperature tar (Burrell, *Oil and Gas J.*, Oct. 10th, 1929, p. 136).

Many patents have been taken out recently concerned with catalytic berginisation of coal; these, approximately 100 in number, are due mainly to the I. G., though the Imperial Chemical Industries and numerous academic investigators have also described several novel features.

Typical working conditions are quoted in E.P. 272,556 (I.G.) which describes the production of low-boiling liquids from coal, oils, tars, etc., by the destructive hydrogenation under high pressure carried out with flowing gases. An example states that crude oil is atomised or gasified in a stream of heated hydrogen *in a vertical high-pressure reaction chamber* lined with aluminium, and is passed at 475° C. and 200 atm. over a catalyst composed of molybdenum and chromium oxides.

The use of wall coatings in the catalyst chamber of such materials that do not catalyse unwanted reactions such as coke or methane formation is a feature of E.P. 302,253 (I.G.), where high anti-knock fuels are claimed by hydrogenation of tars, etc., above 550° C., and with hydrogen under pressure and catalysts such as molybdenum.

The application of catalysts is described in E.P. 307,946 (I.G.) for *one stage* catalytic hydrogenation of coal, tar, etc., to hydrocarbons of low-boiling range. The catalysts are applied to the metallic support in the form of acidified solutions of salts. The metals specified for supports have the following properties and advantages :—

(1) Metals such as aluminium have catalytic properties especially after being etched and catalytic oxides are more active on these etched metals.

(2) Metals must not melt.

(3) Or give rise to carbon.

(4) The *walls* of reaction vessel, pipes, stirring devices, etc., *may be formed of these metals*, and may be coated with catalyst, an additional quantity of which may not then in certain cases be provided. This is particularly an advantage when initial materials are employed containing ash or liable to deposit coke.

(5) Local overheating is less likely to occur owing to heat conductivity of the metals.

These catalysts are particularly suited to the formation of aromatic hydrocarbons, the working temperature being higher than for benzine (500–600° C.).

The examples quoted are specially interesting.

The result of work of the I. C. I. is described in several patents, and E.P. 308,995 of Humphrey and I. C. I. is of interest. The destructive hydrogenation of solid carbonaceous material is carried out in suspension in such an excess of liquid medium of high boiling point that the solid may move freely, a vertical tower being used and hydrogen being employed at 100 atm. or more and at temperatures at 300–550° C. Hydrogen is introduced at the base of the tower and the solid either introduced at the top and allowed to descend by gravity or may be introduced at the base and the additional oil formed withdrawn from the top.

The non-catalytic high pressure destructive hydrogenation of bitumen from tar sands has been patented by Rule, Watts and I.C.I. (E.P. 317,346).

For the purpose of brevity the remainder of the catalytic berginisation patents will be classified under the following headings :—

(*a*) New conditions of temperature, pressure, etc.

(*b*) Additional catalysts.

(*c*) Prevention of coke formation.

(*d*) Elimination of sulphur.

(*e*) Refining of the coal oil.

(*f*) Formation of gaseous olefines, acetylene, etc., from coal tar, etc.

(*g*) General.

(*a*) To select the best conditions for catalytic berginisation, fractions of the raw material of narrow boiling range are treated with hydrogen at high pressure, the process being carried out with the aid of porous catalysts such as charcoal, silicates, hydrosilicates, or active silica (E.P. 273,337 I.G.).

Hydrogenation of coal, tars, etc., in the presence of catalysts, exposing the initial material in thin layers, is mentioned in E.P. 272,190 (I.G.).

A two-stage reaction is quoted in E.P. 272,194 (I.G.) where tars or oils are hydrogenated in the presence of catalysts, and the resulting products in vapour state passed over additional catalysts at higher temperatures. Similarly, E.P. 273,712 (I.G.) states that bituminous materials can be treated first in presence of cracking catalysts with hydrogen under high pressure, and then with catalysts that facilitate hydrogenation. An example is given :—a brown coal tar fraction is injected into a vertical reaction vessel (lined with aluminium), whilst in the liquid state at 450° C. and 200 atm. pressure, and made to flow in counter current with an excess of hydrogen in presence of active carbon. The highly unsaturated products are treated in the vapour state at 450° with cobalt catalyst and thereby hydrogenated. The product is a benzine so stable that further refining is not required. In E.P. 281,247 (I.G.) the destructive hydrogenation of brown coal is stated to give rise to a low value anti-knock fuel, whereas a better value fuel is obtained if the bitumen (benzene-soluble portion of original coal) and wax is removed prior to, or during, treatment. The more intimate mixing of hydrogen and reacting coal, tar, etc., is brought about by passing the hydrogen through porous materials on which the initial material rests (E.P. 272,539 I.G.). The cracking of tars, etc., can be carried out by heating devices immersed in the liquid, the heating elements being exposed to a blast of gases containing oxygen, hydrogen, steam or ammonia (E.P. 295,974 I.G.).

The use of alternating currents to produce viscous oils is described in E.P. 303,776, where such oils, stated to be produced from mineral oils and tar by cracking or hydrogenation in the liquid phase, are subjected to the action of alternating currents of frequencies between 500—10,000 periods/second.

The use of atmospheric pressure in hydrogenation of gases and tar vapours by means of activated hydrogen is claimed in E.P. 275,585 (Prudhomme, Houdry). Bergius (U.S.P. 1,669,439) suggests that when coal is carbonised in gas retorts or coke ovens, the gas can be divided into two fractions and used accordingly: gas poor in hydrogen for towns' gas and the fraction rich in hydrogen for berginisation.

The mention of pressures much higher than the familiar 200–250 atmospheres in catalytic berginisation are made in E.P. 309,239 (Gordon and I.C.I.) where the hydrogenation of coal, tars, etc., to a more uniform product, is claimed by acting upon carbonaceous materials with hydrogen at elevated temperatures and at pressures of at least 1,000 atm. or pressures below (*e.g.*, 200 atm.) and then above 1,000 atm. (*e.g.*, 4,000–5,000 or even 10,000 atm.)

Pre-treatment of coal to remove fusain and durain or partial hydrogenation or oxidation to modify its coking properties is covered by Lander, Sinnatt, and King, in hydrogenation (E.P. 306,564). E.P. 305,744 (Humphrey and I.C.I.) claims a process where coal is crushed and distilled in the presence of oil in such a way that the solid in moving through a continuous body of oil is subjected to progressively increasing temperature. Another modification is described in E.P. 314,213 (I.C.I.), being a process for destructive hydrogenation of solid carbonaceous matter such as coal or lignite of a kind that, on treatment at raised temperature with hydrogen under pressure of the order of 200 atm., a viscous product results. On acting upon this material with hydrogen under pressure of at least 500 atm. at 420° C. a mobile product is formed.

(*b*) As catalysts, combinations of vanadium, manganese, uranium, titanium, chromium, and zinc compounds are claimed (E.P. 257,256 I.G.). Uranium salts are also quoted in the catalytic berginisation of crude cresol (E.P. 295,948 I.G.), and uranium and aluminium in E.P. 295,949 (I.G.). The use of a large excess of hydrogen with catalysts containing boron, phosphorus, arsenic, selenium and tellurium free or combined is claimed in E.P. 299,020 (I.G.), halogens free and combined in E.P. 299,021 (I.G.), and gaseous halogen hydrides in presence of activated metal or alloy in E.P. 303,761 (I.G.). The use of alkali, or alkaline earth, hydrides under pressure, as catalysts in the hydrogenation of tar oils, etc. is covered by Hugel, Paul and Boistel (E.P. 286,206).

Other substances added to increase the efficacy of the reaction are covered as follows :—E.P. 309,227 (I.G.) where tars are cracked in presence of organic substances containing the radicle of a mineral acid such as sulphonic group : E.P. 308,633 (I.G.) solid carbonaceous materials pretreated by heating with alkaline substances before hydrogenation : E.P. 315,991 (I.G.) covers the use of aldehydes and ketones with metals or metallic salts in berginisation of mineral oils, coal tars, etc. : E.P. 309,228 (I.G.) covers the hydrogenation in presence of organic substances containing the radicle of a mineral acid which on admixture with water lowers the surface tension. Ansolvo acid complexes are described as hydrogenating catalysts for coal, tar, etc. : in E.P. 311,789 (I.G.). Peski, (E.P. 315,780) claims the use of pressures below 50 atm. in the destructive hydrogenation, at elevated temperature, of carbonaceous material in the presence of metallic carbides. Florentin, Kling and Matignon (E.P. 279,488) describe a process for the conversion of tars to light hydrocarbons in one stage in the presence of thoria, etc. (dehydrating catalyst), and metal halide (substitutive catalyst) with hydrogen under pressure, the reaction being eminently suited for the conversion of poly-aromatic nuclei-containing bodies and phenolic substances (*cf.* also Kling and Florentin. Brennstoff-Chem. 1927, **8**, 331).

(*c*) The prevention of coke in destructive hydrogenation is described in E.P. 256,964 (I.G.) by the addition of 20 per cent. brown coal or peat, in E.P. 257,910 (I.G.) by the presence of water during hydrogenation, and in E.P. 309,095 (Gordon and I.C.I.) by expediting the hydrogenation with high pressures of the order of 800–1,000 atm. These high pressures are also claimed to reduce the formation of uncondensible gas, and coke formation is also minimised in heating liquid material by moving heating elements (E.P. 272,483, I.G.).

(*d*) Sulphur is eliminated according to E.P. 257,912 (I.G.) by hydrogenating with water present in presence also of catalysts, when the hydrogen sulphide formed is dissolved in the water under high pressure. Lander, Sinnatt and King, (E.P. 302,629) describe a process for taking off a portion of the gases during hydrogenation through a by-pass, subjecting it to treatment to reduce the content of sulphur compounds and other impurities and returning it back to the system.

(*e*) Several novel refining schemes are described for coal oils, such as the use of hydrogen fluoride (E.P. 292,933, Hofmann and Stegemann) and treatment of the benzine and wax freed product of hydrogenation with crude methanol, etc., when tarry oxygenated compounds are dissolved (E.P. 257,270 I.G.). The dilution of

the crude bergin tar with various solvents is described, for example, E.P. 262,738 (Deutsche Bergin-Akt.) states that asphaltic substances in oils from coal can be separated by the addition of petroleum gas oil (*cf.* also U.S.P. 1,709,957 Schoenemann). Watts and I.C.I. (E.P. 311,197) describe a process to effect a ready separation of liquid reaction products of destructive hydrogenation and remaining solid matter by adding powdered coke or coal and diluent oil and filtering hot, and the same patentees (E.P. 311,198) also state that the crude product from destructive hydrogenation can be freed from constituents boiling up to 190° C. and the product diluted with 10 to 50 per cent. of an oil capable of dissolving pitch. Finely divided coal or coke is added to aid filtration. Solvent extraction of the product of hydrogenation is described in E.P. 312,657 (I.G.)

(*f*) The production of gaseous olefines, acetylene, etc., from coal and tars marks a distinct advance in the use of these materials. Thus E.P. 269,593 (I.G.) describes the formation of gaseous olefines, and liquid hydrocarbons by the interaction of steam and tar, etc., in the absence of metals that are attacked by superheated steam and that further the formation of carbon dioxide from carbon monoxide and steam. E.P. 286,825 (I.G.) covers the action of ground coal, etc., in an electric arc in the presence of half or less of water vapour required for water gas (or hydrogen) when acetylene is formed in appreciable amount. Further E.P. 313,756 (I.G.) describes a process where granular bituminous coal is kept in motion by means of a current of vapours and heated by means of an electric current. The vapours which pass over the coal are decomposed, giving ethylene, propylene and butadiene. Tar vapours may also be decomposed by fire brick soaked in iron salts (for conductivity of heat) or granular metal.

(*g*) General processes upon the berginisation of coal, tar, oil, etc., both with catalysts and without are described in the following patents :—E.P. 259,944 (I.G.); E.P. 277,404 (Bianchi); U.S.P. 1,658,116 (Clancy); E.P. 282,814 (I.G.); E.P. 277,419 (Lamplough); E.Ps. 256,965, 286,123, 282,410, 289,170, 272,538, 258,272, 285,923, 286,678, 286,679, 286,680, 286,681, 292,217, 268,796, 270,314, 275,662, 271,451, 293,413, 293,887, 279,072, 275,248, 293,719, 304,343, 294,557, 295,947, 309,229, 311,251, 282,384, 311,628 (I.G.); E.P. 274,465 (Fiege); U.S.P. 1,691,221 (Bernhard and Deman); E.P. 272,526 (Trautmann); E.P. 283,177 (Uhlmann); E.P. 301,949 (Zacon); U.S.P. 1,711,499 (Hofsass); U.S.P. 1,711,856 (Prudhomme); E.P. 299,861 (Girard, Petit and Charboneau); U.S.P. 1,725,320 (Trumble); U.S.P. 1,723,431 (Melamid).

Various reviews and additional experimental papers have appeared upon the subject of motor fuels and oils from coal and

among these may be quoted:—Miltre (*Petr. Zeit.*, **24**, No, 3, p. 103; No. 4, p. 139); Naphtali (*Petr. Zeit.*, **24**, No. 14, p. 611); Varga, "Hydrogenation of an Eocene Peat" (*Brennstoff-Chem.*, 1928, **9**, 277-282); "Conversion of Coal into Oil," Graham and Skinner (*J. Inst. Petr. Techn.*, 1928, **14**, 78-93, and *J. S. C. I.*, 1929, **48**, 129T—136T); Hlavica, "Influence of Mineral Constituents, Especially of Iron Oxide, on the Hydrogenation of Solid Fuels" (*Brennstoff-Chem.*, **10**, 201-203); Aarts, "New Motor Fuel from Coal [Carbon Alpha]" (*Petr. Times*, 1929, **21**, 546, 1,162).

For many instructive papers on the relation of coal and oil in the future, the proceedings of the 2nd International Conference on Bituminous Coal (Pittsburgh), 1928, Vol. I and II, should be consulted.

The technological development (Burrell loc. cit.) of the catalytic berginisation process is now in hand in the United States, and the Standard Oil Company of New Jersey are stated to be building two catalytic berginisation plants whereby cracked residues, it is claimed, will be converted into 100 per cent. gasoline, using pressures of 3,000 lbs. sq./in. and upwards.

It should be noted that with hydrocarbon oils these high conversion yields are possible whereas with coal, especially the younger coals, the oxygen, nitrogen and sulphur content materially lowers the yield of hydrocarbons.

A further report (*Oil News*, July 19, 1929, p. 54) states that three plants of 5,000 barrels capacity are being built at Bayway, N.J.; Baytown, Texas (Humble Oil and Refining) and at Baton Rouge, Lousiana. This account accentuates the evolutionary rather than revolutionary development of petroleum refining in this direction and suggests that catalytic berginisation will not lead to the disuse of present-day plant but must be regarded as possible supplementary plant. The subject is full of potentialities in the future, considering the amounts of low gasoline containing crude oils available for example from Venezuela.

Synthetic Liquid Fuels, Olefines, and Acetylene, from Hydrocarbon Gases, Water Gas, Etc.

Previous to 1928 it had been established that ethane and its higher homologues could be treated at temperatures about 600°C. and higher, to give either the corresponding olefine by loss of hydrogen or a lower olefine by loss of methane, etc., and at temperatures higher than this to form aromatic hydrocarbons such as benzene.

The production of olefines by such thermal dehydrogenation is reported (Elworthy, *Ind. Chemist*, 1928, **4**, 275).

Methane was regarded generally as being not amenable to pyrolysis until Fischer (*Brennstoff-Chem.*, 1928, **9**, 309) showed

that under very limited conditions of high gas speeds and at temperatures of 900-1,100°C. methane can be cracked to yield 12-13 per cent. of higher hydrocarbons chiefly of the aromatic series. These results were substantiated by Stanley and Nash (*Nature*, 1928, **122**, 725, and *J. S. C. I.*, 1929, **48**, 1T), who obtained a 20 per cent. conversion of the methane into liquid and gaseous hydrocarbons higher than the original methane. Similar results are recorded by Wheeler and Wood (*Fuel*, 1928, **7**, 525), and the mechanism of the general thermal decomposition of n-paraffins is exhaustively treated by Hague and Wheeler (*J. C. S.*, 1929, 378-391). Similarly the thermal decomposition of ethane, ethylene, propane and propylene has been described by Frey and Smith (*Ind. Eng. Chem.*, 1928, **20**, 948-951).

The thermal decomposition of gases containing paraffins into aromatic hydrocarbons has been patented by Wheeler, Dunstan and A. P. O. Co. (E.P. 309,455) and with special reference to methane by Fischer (E.P. 319,340). Catalysts such as chromium, zinc, aluminium, their oxides and manganese chromate are mentioned in a similar process for the manufacture of aromatic hydrocarbons from non-aromatic hydrocarbons in the presence of hydrogen by the I. G. (E.P. 281,298).

Several patents have been taken out for the production of olefines and acetylene. Thus E.P. 264,827 (I. G.) covers the production of ethylene and acetylene by passing aliphatic hydrocarbons (especially methane) over catalysts such as the salts of the alkaline earth metals, and E.P. 301,402 (I. G.) covers the formation of unsaturated hydrocarbons of the same number of carbon atoms by passage of hydrocarbons other than methane over heated catalysts.

In E.P. 264,845 (I. G.), methane is stated to be converted into acetylene by passing the gas diluted with nitrogen through a disc like flame at high temperature.

The production of olefines and diolefines is covered by E.P. 303,998 (I. G.) where the paraffin vapours are brought into contact with such catalysts as lustrous carbon; butadiene is among the unsaturated bodies mentioned in E.P. 307,945 (I. G.), in a similar cracking process. The use of electrical discharge in the transformation of gaseous hydrocarbons is described by Hull and I. C. I. (E.P. 304,914) and by the I. G. (E.P. 315,249).

The conversion of methane by means of an electric discharge has been shown by Stanley and Nash (*J. S. C. I.*, 1929, **48**, 238T) to give rise to tarry products and the lighter oils from these, instead of being aromatic in nature (as those from the thermal decomposition), are very unstable and resinify on standing. The chief product of the reaction is acetylene. High yields of acetylene are claimed by the I. G. (F.P. 626,795) by this process. A short review of this

process is included in a recent paper by Stanley (*J. Inst. Petr. Techn.*, 1929, **15**, 534).

Patents relating to the plant required for working with hydrocarbons at high temperatures where soot and coke forming metals are to be avoided are E.Ps. 296,683 ; 296,693 ; 296,694 ; 296,752 ; 296,753 (I.G.).

The polymerisation of acetylene has been investigated by Bangert and Pichler (*Brennstoff-Chem.*, 1929, **10**, 279-282) who find that on passing acetylene over activated charcoal or silica gel at 600-700°C, hydrogen, methane and carbon are formed in the first instance, the latter carbon, however, acts as a condensation-catalyst later in the formation of liquid hydrocarbons. The oils which contain equal portions of light and heavy fractions contain aromatics (preponderating) and unsaturateds in the light spirit.

Further experiments are given by Fischer, Peters and Koch (*Brennstoff-Chem.*, 1929, **10**, 382-385) where acetylene and hydrogen are passed over a catalyst of iron-copper and alkali at 250°C when 40-70 per cent. of the acetylene is converted into oil.

The polymerisation of olefines is again a subject of prime importance. Liquid hydrocarbons from heat treatment of olefines under pressure in presence of gold, silver, copper and alkali metal salts are covered by E.P. 291,137 (I.G.). At 300-400°C liquid olefines are formed and at 550°C the bulk of the products are benzene and naphthalene hydrocarbons.

Viscous oils are produced by treating olefine gases with hydrocarbon mixtures containing aluminium chloride (E.P. 299,086 I.G., c.f. also E.P. 309, 199, I.G.).

The acceleration of the polymerisation of olefines by means of boron trifluoride with traces of moisture and halogen acids is described by Hofmann Otto and Stegemann (E.P. 313,067).

The action of a silent discharge upon hydrocarbons to give liquid power fuels (E.P. 294,100) and lubricating oils (E.P. 294,099) is covered by Epner.

The production of liquid fuels, olefines, etc., from carbon monoxide and hydrogen mixtures has also been the subject of much technological and academic research.

The production of olefines with paraffin hydrocarbons by the catalytic interaction of carbon monoxide and hydrogen mixtures have been described by Nash, Bowen and Elvins (E.P. 291,867) and I.G. (E.P. 293,572). The latter firm have also covered the formation of liquid hydrocarbons in the presence of certain catalysts (E.Ps. 269,521 ; 293,185 ; 271,452 ; 312,717).

The production of benzene is claimed from water gas E.P. 297,179 (I.G.), however, not directly, but by cracking the first stage gases from catalytic action at temperatures between 600-800°C.

Experimental details of work upon this subject are given by Smith, Davis, Hawk and Reynolds, "Synthesis of Higher Hydrocarbons from Water Gas, Pt. I. and II.," (*Ind. Eng. Chem.*, 1928, **20**, 462-464, 1341-1348).

The synthesis of alcohols from carbon monoxide and hydrogen appears to be more closely related to the chemical than petroleum industries. The following contributions to the scientific and patent literature have been made during the last two years :—

Methanol-E.P. 293,056 (Synth. Am. and Nitrates and Franklin ;) Frolich, Fenske, Taylor, and Southwich (*Ind. Eng,. Chem.* 1928, **20**, 1327-1330) ; Brown and Galloway (*Fuel*, 1928, **7**, 518-525) ; Smith and Branting (*J. Amer. Chem. Soc.*, 1929, **51**, 129) ; Cryder and Frolich (*Ind. Eng. Chem.*, 1929, **21**, 867-871) ; E.P. 272, 864 (Commercial Solents Corp.).

Dimethylether—Brown and Galloway (*Ind. Eng. Chem.*, 1929, **21**, 310).

Higher alcohols—Frolich and Lewis (*Ind. Eng. Chem.*, 1928, **20**, 354-359).

General considerations—E.P. 313,467 (I.G.) ; Frolich, Fenske, and Quiggle [catalysts and thermodynamics] (*Ind. Eng. Chem.*, 1928, **20**, 694-7) ; Frolich [role of catalysts] (*J.S.C.I.*, 1928, **47**, 173-9T).

Several miscellaneous patents have been issued covering new processes for the production of liquid hydrocarbons, olefines gas mixtures, alcohols, etc. Liquid hydrocarbons are claimed by heating metallic carbides in presence of hydrogen (E.P. 270,705, I.G.), and unsaturated hydrocarbons by partial combustion of liquid hydrocarbons (E.P. 290,322 I.G.). E.P. 274,904 (I.G.) refers to catalytic gas reactions ; E.P. 294,787 (I.G.) to ethylene production from acetylene and hydrogen ; E.P. 302,297 (I.G.) to the recovery of organic solvents ; E.P. 302,203 (I.G.) to the separation of gaseous and low-boiling hydrocarbons from gases such as cracked gas ; E.P. 301,969 to formation of carbon monoxide and hydrogen from hydrocarbons ; and E.P. 303,323 (I.G.) to the manufacture of diolefines. Potts (E.P. 309,002) claims the synthesis of liquid hydrocarbons by the action of an intense beam of beta rays on gaseous carbon compounds.

The production of carbides (that give acetylene on treatment) from methane, and metal oxides has been investigated by Fischer and Bangert (*Brennstoff-Chem.*, 1929, **10**, 261), cf. also Peters and Meyer (*Brennstoff-Chem.*, 1929, **10**, 324).

Finally, there is the work of Fischer and Pichler (*Brennstoff-Chem.*, 1928, **9**, 200-206) describing the formation of gaseous and liquid hydrocarbons by the action of steam and alkali on semi-coke at increased pressure, and E.P. 301,000 (I.G.) where iso-propyl, butyl and higher alcohols are claimed to be obtained from the products of destructive hydrogenation of coal and oil by dehydrogenation and the addition of water to the double bond. Both of these reactions serve to indicate the diverse paths of recent research and that the production of liquid fuels and chemical intermediates are becoming more closely related.

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Analysis and Testing*

By C. I. Kelly, M.Sc., F.I.C., F.C.S. (Associate Member).

During the past year the Institution of Petroleum Technologists has issued the Second Edition of Standard Methods of Testing Petroleum and Its Products, in which new classes of products have been included.

Class III. Benzole Mixtures.—Methods for these are for Specific Gravity, Colour, Distillation, Total Sulphur, Acidity and Aromatic Content. A notable exclusion is that of a method for corrosion or free sulphur, but when necessary the method given for Gasolines could be applied.

Class IV. Long-Time Burning Oil.—The methods given deal with Specific Gravity, Colour, Distillation, Total Sulphur, Flash-Point, and Burning Test. In the last named, the Welch Lamp No. H.S. 68 or the No. 5 " Adlake " Semaphore Lamp may be used.

Many additional methods and modification of apparata have been included in the other groups.

Class I. Gasoline.—The distillation test has been brought into line with the A.S.T.M. Standard Method.

In addition to the well-known Lamp Method for the determination of total sulphur, the Richardson Method has been included as an alternative. The apparatus, Pat. No. 211,925, has several lamps—one for each class of products (gasoline, benzole mixtures, and kerosine). The method has several commendable features: (1) It burns the products rapidly and with a non-luminous flame; (2) The estimation is titrimetric. On the other hand, the apparatus is expensive, and this is particularly so when it is compared with that used to estimate sulphur by the almost equally rapid method of Squire.[1] Although not a standard method, this method can be worked up in individual laboratories for internal use to give results which compare very favourably with most accepted standard methods.

The Copper Dish Corrosion Test has been displaced, and a method for the Estimation of Elemental Sulphur has been added. This method is based on that originally put forward by Ormandy and Craven,[2] and since then elaborated by Hoffert and Claxon.[3] The method has appeared in a slightly modified form as a Bureau of Mines publication,[4] and another modification has been worked

* Paper received Feb. 8, 1930.

out on the Continent.[5] The present method for the estimation of aromatic hydrocarbons does not provide for the estimation of the individual aromatics, as was the case in the older method.

Class II. White Spirit.—For this product a new method provides for the detection of reactive sulphur, whilst there is no longer a method for detecting oily matter.

Class IV. Kerosine.—Under flash-point (K.2), a method has been added for the determination of the Abel flash point of solids which may contain inflammable constituents.

Class VI. Gas Oils.—The Conradson method for carbon residue has been altered slightly. A wire bridge 2 in. high is fixed on top of the chimney, as a guide to the height of the flame during the burning period. Also two small glass beads 0·1 in. in diameter have to be placed in the crucible (before weighing it empty) in order to promote more regular ebullition of the oil. The duration of the pre-ignition, burning and final heating period are more clearly defined than formerly. As an alternative to this method, one can now use the Ramsbottom method, which has been in use for many years in Government laboratories.

Class VII. Lubricating Oils.—In addition to the Redwood viscometers, one may employ the U-tube and coaxial bulb types of viscometers (which have been standardised by the British Engineering Standards Association), and especially when absolute viscosities are required. In view of this, attention is drawn to the publication of a revised edition of the B.E.S.A. Specification No. 188 in 1929 (22 pages, price 2s.). The ten appendices of this more explanatory edition deal with the quality of the glass and annealing, Poisseuille's Law, notes on method of carrying out an observation, the viscosity of water at various temperatures, the preparation and viscosity tables of 40 per cent. and 60 per cent. sugar solutions, etc. References to original papers are also given as footnotes. Because the various sizes of viscosimeters are recommended for the measurement of viscosities of gasoline and kerosine as well, this publication is, in effect, a companion of the new book of standards.

A new method of test for determining the setting point of lubricating, gas and fuel oils replaces the old Pour Point Test. It involves the use of a U-tube 19 mm. in internal diameter, with right-angled bends.

Class VIII. Transformer and Switch Oils.—The methods for the determination of volatility, sludging value and dielectric strength differ from those appearing in the first edition. A larger volume of oil and a shorter heating period are used in the volatility test, now, and 100 grams of oil, not 100 mls., are to be used in the sludge test.

The dimensions of the condenser fitted to the sludge flash have been completely altered. The specification of the petroleum spirit with which the oxidised oil is to be diluted has been extended to include limits for the iodine value (Hubl), the critical solution temperature of the acid treated spirit, whilst a maximum of 2 per cent. of aromatic hydrocarbons is now permissible.

Class IX. Fuel Oils.—The old method for hard asphalt has been modified to restrict the amount of oil taken, so that the weight of hard asphalt obtained shall not exceed 0·25 gram.

Class X. Asphaltic Bitumens and Asphaltites replace the old Class VIII. asphalt. Methods for the determination of asphaltenes (hard asphalt) and the determination of matter insoluble in carbon disulphide are specified now for these products. On the other hand, the method for the determination of wax has been excluded and preference is given to a new method for the determination of asphaltum (soft asphalt). The penetrometer needle's dimensions have been altered and a few alterations have also been made in the method for the determination of the softening point (ring and ball method).

Class XI. Commercial Paraffin Scale and Paraffin Wax replaces the old Class IX wax. New methods are included to cover the determination of colour, moisture, dirt, and expressible oil, whilst the method formerly appearing under the name of melting point has been included under the heading of setting point.

Class XII. Crude Oil.—Additions to this section include methods suitable for the dehydration of the crude oil, large scale distillation, whilst the methods for the determination of ash, hard asphalt, soft asphalt, and cold test have been excluded.

Sampling.—At the end of the book there is a chapter on sampling. Opportunity is taken here of emphasising the harmful nature of the practice of pouring sealing or paraffin wax over the corks of sample bottles, and also the necessity of protecting samples of turbine, medicinal, and transformer oils from contamination, by using ground glass stoppered bottles. Over-emphasis of these points cannot be made since the accuracy and validity of laboratory results depend entirely upon the purity of the sample received for examination.

British Engineering Standards Association.

During the period under review, several B.E.S.A. specifications containing methods of testing sometimes required by petroleum laboratories have either been revised or newly issued. No. 76-1930, Tars (Nos. 1 and 2) for Road Purposes. No. 188-1929,

Determination of Viscosities in Absolute Units. No. 342-1928, Two Coat Asphalt (Sand or Sand and Stone Aggregate Wearing Surface). No. 343-1928, Two Coat Asphalt (Clinker Aggregate). No. 344-1928, Single Coat Asphalt (Sand and Stone Aggregate). No. 345-1928, Single Coat Asphalt (Clinker Aggregate). No. 346-1928, Mastic Asphalt Surfacing. No. 347-1928, Asphalt Macadam (Penetration Method). No. 348-1928, Compressed Rock Asphalt.

Bureau of Mines, U.S.A.

Technical Paper No. 323-B, U.S. Govt. Specification 2D, United States Govt. Master Specification for Lubricants and Liquid Fuels (and Methods of Sampling and Testing), has been issued by the above body, and became effective on January 21, 1928.

Tar Products.

In December, 1929, an important publication appeared as a result of a resolution passed at the Tar Conference at Manchester in 1926. The meeting gave rise to the formation of a Standardisation of Tar Products Tests Committee (166 Piccadilly, London, W. 1). This body has issued a book of "Standard Methods of Testing Tar and its Products" (291 pages, 7s. 6d. net). In it will be found methods of testing for crude tar, refined tar including road tar, lower boiling fractions (light oil and crude benzole), benzole fore-runnings, benzole, naphthalene, tar bases, creosote oil, crude anthracene and pitch.

American Society of Testing Materials.

During 1928 several new tentative methods were published: (1) Definitions of Terms Relating to Petroleum (17 terms were defined). (2) Test for Distillation of Crude Petroleum (300 c.c. are distilled in a Hempel distillation flask). (3) Test for the Determination of Autogenous Ignition Temperatures (based on work carried out by the sub-committee on four types of apparata, including that of Moore[6]). (4) Test for Gravity of Petroleum and Petroleum Products by means of the Hydrometer.

Several tentative methods were advanced to standard: (1) Test for Saponification Number. (2) Test for Cloud and Pour Points of Petroleum Products. (3) Methods of Testing Gas Oils (gravity, distillation, sulphur, carbon residue, pour point, viscosity, water). (4) Test for Carbon Residue of Petroleum Products (Conradson carbon residue).

In 1929, several tentative and standard methods were modified and also there was published a new tentative method for the determination of expressible oil and moisture in paraffin wax.

The various sub-committees have continued their activities, and some of their findings are :

(1) The Tentative A.S.T.M. Method for Melting Point of Petrolatum is preferred to the Ubbelohde Method.

(2) More value is attached to the Tentative Method for Expressible Oil and Moisture in Paraffin Wax than to other methods such as Solvent Index of Refraction, Solidification Method, and Direct Refraction Method.

(3) In the Test for Precipitation Number of Lubricating Oils (equivalent to Hard Ashphalt Content in this country), provided that the aniline point of the Precipitation Naphtha is between 58°C and 60°C. little trouble will be found in getting good agreement between laboratories checking each other on the same oils, but using naphthas from different crudes. The boiling range is not a major factor, and the Final Boiling Point can lie between fairly wide limits.

(4) Steps are being taken to estimate the value of an Oxidation Test at High Temperatures (Sligh Method) as a means of valuating Turbine Oils.

(5) The Fuel Oil Committee stresses that the selection of a Fuel Oil should be based on Viscosity and not Gravity. It hopes also to devise a test which will indicate whether oils from different sources can be combined in storage without precipitating sediment.

(6) It is hoped shortly to recommend a Tentative Method for the Determination of the Dilution of Used Crankcase Oils.

Testing of Gasolines in Engines for Knock-Rating.

During the last few years and particularly in the period under review, rapid progress has been made in the commercial practice of testing gasolines in engines. The Ethyl Gasoline Corporation has experimented with numerous types designed for this purpose and recently accounts of some of them have appeared in the current literature.[7] Descriptions and illustrations have been given of (1) the present type of Ethyl Gasoline Corp. Detonation Testing Engine (which is used in several laboratories in this country) ; (2) an air-cooled Detonation Testing Engine ; (3) A large Bore Engine, and (4) a variable compression Engine, all of which are used by that body. An account is also given of the variables which have a bearing on the relative ratings of fuels in Automobile Engines. Conclusions are also drawn from a programme of testing (not then completed) which support the contention that there is no justification for building an engine whose cylinder is a duplicate of an

automobile or aero engine cylinder, in order to rate fuels properly. Conditions can be found for a given engine under which one can rate fuels in the order of merit in which they fall when tested in conditions of service. In conclusion are enumerated the features which should be found in an ideal knock testing engine.

The variable compression engine mentioned was more fully described later.[8] This unit has been studied jointly by the American Petroleum Institute, American Society of Automotive Engineers, Bureau of Standards, etc., and is being put forward as a Standard Knock Testing Engine. It is ruggedly built : it can be converted from an ell head unit into a variable compression engine : it is suitable for the throttle or spark method. (A neon tube spark indicator is fitted behind a quadrant graduated in degrees) : provision is made for warming up the lubricating oil before starting : it can be worked at a uniform jacket temperature and it has a special plate orifice jet in an Ethyl Gasoline Corporation carburettor which provides a uniform air-fuel ratio over a fair range of loads, and provision is made for automatic maintenance of uniform engine speed. The bouncing pin can be used or detonation tests can be carried out by the Listening Method. When the latter is used, differences in ear sensitivity among operators are overcome by producing a "standard sound." The sound box and a listening post in the cylinder head are connected through a sort of stethoscope applied to the ears. Intensity of detonation is regulated against this sound.

The results of a study of the measurement of Detonation carried out in the Air Ministry's Laboratories in conjunction with the E.G. Corporation and the Anglo-American Oil Co. have been reported in this country during the past year.[9] Descriptions of the three classes of methods of measurement of Detonation are described and an analysis of the Bouncing Pin Apparatus and Method is carefully worked out alongside that of the measurement of the H.U.C.R. in Ricardo's Variable Compression Engine (E. 35 Type).

The Standardisation of Conditions for Measuring the Detonation Characteristics of Motor Fuels has been studied in the Anglo-Persian Oil Co.'s Research Station.[10] In this report three engines are described : (1) a Thornycroft Variable Compression Engine ; (2) a Ricardo Single Sleeve valve Engine ; (3) a modified single cylinder "B" Type Armstrong-Whitworth Engine. The experimental procedures used on these engines are given in detail together with an estimation of the Experimental Error, a discussion of the influence of controllable variables, Fundamental Standard Fuels, etc.

Accounts of the Knock Testing engine used by the Pierce Petroleum Corpn., Oklahoma, has also been described in the literature.[11]

Several important papers have been published recently which deal with the comparison of methods of measuring Knock Characteristics of fuels.[12] [13] [14] Attempts have also been made to correlate the measurement of anti-knock values of gasolines in ten laboratories, with a view to determining the effect of volatility on these values.[15] It has been reported that accuracy in measuring detonation arises out of the giving of proper consideration to mechanical variables in the engine.[16] Another contribution describes the audible spark advance method of testing gasolines in engines [17] and elsewhere, it is stated that the use of mixture strengths giving maximum knock is a means of getting consistent results from different laboratories.[18]

Estimation of Gum in Gasolines.

During the last two years the question of the presence of gum in gasolines has been widely discussed, and the necessity for methods of gum estimation has arisen in consequence. Careful distinction has been drawn between the gum found in the product at a given time (preformed, existent or actual gum) and the gum which is likely to be formed in storage after that time. (Potential gum.)

The estimation of preformed gum.—The copper dish test has been criticised adversely by many and appraised by a few. Suggestions have been made that the evaporating dishes should be made of silica or glass and that the evaporation should be carried out in an inert atmosphere (steam), or on baths with a controlled air blast directed on to the surface of the evaporating liquid.[19], [20] On the other hand, some of the methods advanced, aim at the reproduction of intake manifold vaporisation. In one of these, the gasoline is dropped slowly into a heated glass helix through which air or nitrogen is being aspirated. The increase in weight of the glass spiral is due to the gum deposited during the flash evaporation.[21] In another, a gravity-fed carburettor sprays the gasoline into a system containing a small hot plate on which the gum particles collect.[19]

The estimation of potential gum in/or stability of the Gasoline.—The methods so far developed, have been of the nature of oxidation tests carried out at temperatures which are high, relative to those of storage conditions. In one[22], the gasoline is placed in a glass flask from which the air is expelled and replaced by oxygen. The corked flask is kept at 100° C. for several hours and then the gasoline is evaporated in a glass dish in an inert atmosphere. Such oxidation tests are affected like many others, by very small and seemingly unimportant details. For instance, it has been shown that the use of rubber corks in these glass flasks markedly

affects the characteristics of the absorption curves.[23] In another a glass bottle containing the gasoline is placed in a steel bomb which is charged with oxygen to a pressure of 100 lb. per sq. in. The bomb is maintained at 100° C. in a boiling water bath while a time-pressure curve is being drawn on a distance-recording instrument. The stability of the gasoline is considered to be proportional to the length of the induction period (the time which has elapsed when a fall in pressure is observed).[19] It is interesting to note that an oxygen-bomb method similar to this, has been of much service to rubber chemists in their endeavours to produce rubber articles of good ageing qualities, because by means of this test they are able to carry out rapid artificial ageing tests on experimental "mixes."[24] The estimation of potential gum in motor benzols has been investigated along the lines of bubbling air or oxygen through cold or hot benzol under reflux.[25 26]

Analysis of Cracked Gasoline.

The analysis of cracked gasolines has continued to receive attention although the workers abroad, still seem to be unaware of the limitations of sulphuric acid for this purpose. A new departure has been taken in the use of fuming nitric acid[27] for the removal of the aromatic and unsaturated hydrocarbons simultaneously and at a low temperature. Naphthenes and paraffins are estimated on the unattacked residue whilst the unsaturated hydrocarbons are estimated by means of the iodine value of the original spirit. Full working details together with a nomograph for use in the estimation of iodine values, have been published. Methods for the determination of the composition of kerosines[28] and gas oils[29] have also appeared in the current literature during the last two years.

Lubricating Oils.

In the case of lubricating oils, no serious work of a similar nature has been described, but advances have been made in perfecting the routine methods available for the testing of these products. Whilst the Conradson carbon residue method has been made more useful, the standardisation of the Ramsbottom method has placed the industry in a position to estimate this feature of lubricating oils with greater precision. The method has been discussed in this journal during the past year.[30]

Viscosity.

Efforts are being made in the States to make it possible for laboratories to standardise their Saybolt viscosimeters. In this country, the standardisation of Redwood viscometers is carried

out by the National Physical Laboratory where viscosimeter " X " is compared with the standard instrument by means of an oil, the viscosity of which (about 300 secs. at 70° F.) is determined on both instruments at room temperatures. In the States, the Bureau of Standards check the dimensions of the tube and jet. Recently, supplies of two oils have been reserved. One oil is used to standardise the Universal viscosimeter at 100° F. and the other is used both on the Universal at 210° F. and the Furol at 122° F. Supplies of these oils of given viscosity may be obtained from Dr. Delbridge of the Atlantic Refining Co. or from The Bureau of Standards, U.S.A.

Another development during the last two years which deserves special mention is that the American Chemical Society has started the publication of analytical methods in collected form in the Analytical Edition of the *Journal of Industrial and Engineering Chemistry*.

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A.P.I. Research Programme.*

By GEORGE SELL (Member).

THE American Petroleum Institute's programme of scientific research in the geology, physics and chemistry of petroleum reached the end of its third year on June 30, 1929. Brief statements of progress[1] in the various projects of research are issued annually and the following information has been extracted therefrom. The whole of this research is financed from funds contributed by Mr. John D. Rockefeller and the Universal Oil Products Company.

GEOLOGY.

Project No. 1 is research into the *Generation of Oil in Rocks by Shearing Pressures*. Laboratory experiments to duplicate natural conditions would necessitate the combination of all, or nearly all, of the possible variable factors. Geologic time cannot be duplicated and high temperatures and pressures present difficulties, so it was decided to determine the separate effect of two variables at a time. Shearing pressure tests over varying periods were negative and the heat of friction developed was insufficient to have measurable effects on the oil-shales. Using catalysts, with or without shearing pressures, anhydrous aluminium chloride had a distinct effect on the organic matter, increasing its solubility in organic solvents. Absorbents decrease the solubility of the extractible matter.

Extraction with chloroform gives a greater extraction from the sheared than from the unsheared specimens.

Work is proceeding on the effect of combined pressure and heat on oil-shales.

The Studies of Source Rocks in the Microfurnace, Project No. 3, is intended to secure information as to the origin of petroleum and to throw light on the amounts and qualities of oil produced artificially from the various components of the "mother rocks" and the temperatures at which they are yielded. This bears directly on the production of petroleum substitutes from oil-shale and coal.

The tests have confirmed the basic conclusion that many of the visible organic components, although changed in composition, are chemically distinguishable until the organic sediments have

* Paper received January 31, 1930.

[1] *Bull. Amer. Petr. Inst.*, 1927, **8**, No. 54; 1928, **9**, No. 53; 1929, **10**, No. 55.

reached advanced stages of natural carbonisation. The structureless biochemical matter of the groundmass colloid offers a wide range in composition and probably embodies the organic substances, acids, etc., remaining from the soft parts of animal organisms initially contributed to the sedimentary deposit.

In the "fatty" algæ, the cell contents react at temperatures different from those of the fatty lining of the cell wall, showing chemical difference.

Since petroleum source rocks intergrade into coaly sediments and coals, the conclusion is justified that the chemical distinctions of organic components in the coaly series similarly persist.

The microthermal studies of a group of Alaskan source rocks have been reported on.[2]

The conditions under which possible oil-yielding sediments are now being formed is being investigated under Project No. 4, *Origin and Environment of Source Sediments*. Deposits from a large number of localities have been obtained and the richest deposits studied in detail.

Analyses have been commenced on four of the richest deposits, which were dried, ground and extracted with carbon tetrachloride. The extracts contained fatty acids from 2 to 16 parts per 100,000, organic sulphur compounds up to 36 parts, pigments about 5, sterols less than 1 in the marine, but 18 in an algal lake sample, and solid paraffins up to 16 in the marine and 68 in the algal lake deposits. Free sulphur comprised 22 to 104 parts per 100,000.

It is inferred that liquid petroleum forming in sediments during their accumulation cannot be a major factor in commercial oil pools, but the presence of solid paraffins indicates that some petroleum constituents are present in newly deposited sediments. Analyses of the remaining organic matter are being carried out.

Diatoms as a Source of Oil are being investigated under Project No. 5. Chemical examination of the products extracted from living diatoms by fat solvents shows the presence of small quantities of hydrocarbons. Large amounts of fatty acids, fats and soaps were also encountered and sterols are present. Besides the yellow and green plant pigments, manganese- and zinc-containing pigments were also extracted.

Biological research has been chiefly concerned with the diatom *Aulacodiscus Kittoni*, Arnott, but all efforts to culture this organism have failed. The occurrence of a mass of *Aulacodiscus* near Corinta, Nicaragua, strengthens the belief that there is no reason to exclude the tropics as an autonomous oil-producing area.

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[2] *Bull. Amer. Assoc. Petr. Geol.*, 1929, **13**, 823–840.

Bacteriological investigation of the ærobic decomposition of oils and fats did not give any important results and the continuation of work on the anærobic phase only seems justified.

Experimental work on limestones under Project 23, *Limestones and Dolomites as Reservoir Rocks*, has shown that limestone absorbs the lighter fractions of the oil, while the heavier fractions tend to adhere to the rock. In finely powdered limestone, the oil is displaced by water. Tubes filled with oil-saturated crushed limestone were flooded and gave 76·7 per cent. recovery with water, 80·9 per cent. with a deci-normal solution of sodium carbonate, and 83·8 per cent. with a deci-normal solution of acetic acid. The average recovery by continuous floodings was 70·3 per cent., as against 81 per cent. by intermittent flooding. It was concluded that if the water advance was retarded, the total recovery from a limestone reservoir may reach 85 per cent. of its oil content, the remainder being unrecoverable even by mining methods.

The *Structural Relations of Beds that are Separated by Converging Strata* has been dealt with under Project No. 24, and it seems that convergence along the surface of unconformities is an important factor in the localisation of folds. A definite relationship is indicated between the axial plane of folds and the direction of convergence and the disposition of folds is apparently determined by the difference in strength of layers.

Physics.

Under Project No. 6, *The Separation, Identification, and Determination of the Chemical Constituents of Commercial Petroleum Fractions* has been investigated and a paper describing apparatus and methods has been published.[3]

A gas, an "ethane," a "propane," a "normal butane," and a "mixed butane" fractions have been isolated, and four of the isomers of hexane have been isolated and their principal properties determined.

It has been discovered that heavy hydrocarbons can be distilled without cracking from surfaces having temperatures over 1000° C.

Catalytic Methods applied to Petroleum Hydrocarbons, Project No. 7, have received considerable attention. Equilibrium in the reaction $C_2H_6 \rightleftarrows C_2H_4 + H_2$ has been measured at 600°, 650° and 700° C.[4] and indicates that the constants are 0·031, 0·082 and 0·20 respectively.

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[3] *Bur. Standards Res. Journal*, 1929, **2**, 467–488.

[4] *J. Amer. Chem. Soc.*, 1928, **50**, 2715–2718.

The reactions of mixtures of oxygen with propane and the butanes[5] are classified into three types, (1) unsaturates by dissociation, (2) unsaturates by oxidation, and (3) aldehyde. The first two appear to be normal gas reactions at 500° to 600° C., subject to induction by type 3 at lower temperatures. Type 3 may begin at 300° to 350° C. and is indicated as a chain reaction. Under suppression, no reaction may occur up to 500° to 600° C., then types 1 and 2 appear, accompanied by type 3.

In the thermal dissociation of the butanes, of the reactions

$$C_4H_{10} \rightarrow C_4H_8 + H_2 \quad (1)$$
$$\rightarrow C_2H_6 + CH_4 \quad (2)$$
$$\rightarrow C_2H_4 + C_2H_6 \quad (3)$$

only (2) and (3) are of importance in the case of *n*-butane, and (1) and (2) in the case of iso-butane.

The reaction in the polymerisation of acetylene is a homogeneous, bimolecular gas reaction. No reactions other than polymerisation are of importance up to 650° C.

Ethylene, on polymerisation, appears to be completely converted to a butylene between 450° C. and 600° C., and at about 600° C. it also gives rise to ethane. Hydrogenation can compete with polymerisation at about 600° C.

The reaction of hydrogen-oxygen in Pyrex is in part conditioned by catalytic action on the surface, which is decreased by treatment with hydrogen at 600° to 650° C., and further by coating the glass with potassium chloride or hydroxide.

In the *Analysis of the Gradual Oxidation Prior to Ignition of Fuels in Internal Combustion Engines and the Relation of such Oxidation to Detonation*, Project No. 11, the effect of lead tetraethyl on compression pressure was measured to see if the greater oxidation of a non-doped charge was accompanied by an increase in temperature. No appreciable difference was observed and it was assumed that oxidation (1) takes place on the surface, or (2) occurs prior to the start of compression upon the first contact of the charge with hot walls and residual exhaust.

The addition of the tarry condensate, collected after oxidation in heated tubes of a rich air-hexane mixture, to a one-ether two-acetone mixture did not change the intensity of the knock. The peroxide content was 5·6 g. per litre, and the result contrary to the peroxide theory of knocking.

Tubes coated with lead oxide reduced oxidation more than lead tetraethyl vapours. The hypothesis is advanced that, in the presence of lead oxide, the hexane molecule upon initial oxidation remains less activated, while, when lead is absent, the hexane

[5] *J. Amer. Chem. Soc.*, 1929, **51**, 1839–1856.

molecule continues to oxidise in the gaseous phase until all hydrogen is oxidised.

In the *Determination of Geothermal Gradients in Oil Fields on Anticlinal Structure*, under Project No. 25, a large number of wells in various fields have been tested.

In Oklahoma the lowest gradient was at Healdton (1° F. in 149 ft.) and the highest in Glenn Pool (1°F. in 36½ ft.).

In the Humble Salt Dome, Harris County, Texas, it was found that (1) at equal depths temperatures are higher on the top than on the flanks of the dome, (2) temperature increase with depth is greater on the top than on the flank, (3) this difference and greater gradient in temperature are found not only near the salt but also above the dome, and (4) borings on top of salt domes have approximately the same temperatures at equal depths.

In the Santa Fe Springs field of California, shallow temperatures vary very widely, being between 65·9° to 74·9° F. at 100 ft. The reciprocal gradients over depths of 100 to 2,000 ft. vary from 47·5 to 56·6 ft. per degree. In the Long Beach field the variation in reciprocal gradients is about 8 ft. per degree, the mean isothermal depth being 1,534 ft. and the mean reciprocal gradient about 53 ft. per °F.

The object of Project No. 26, *Determination of the Thermal Conductivity of Gases and Its Application to the Precise Analysis of Hydrocarbons and Other Gases*, is to obtain a definite measure of the behaviour of an electrically heated wire in a bath of gas under known conditions. It is proposed to study the whole series of hydrocarbons and other gases for their characteristic values, and then to examine their comparison with a two-cell or multiple-cell comparator relative to known gas mixtures.

In order to study *The Displacement of Petroleum Oils from Oil Bearing Sands by Use of Selected Aqueous Solutions*, Project No. 27, it was necessary, as crude oils are too complex for the preliminary work, to use some simple binary systems. Liquid-liquid interfacial tensions[6] and adhesion tensions were measured for five such systems: alpha bromnaphthalene-benzene; benzene-ethyl carbonate; alpha bromnaphthalene-ethyl carbonate; dimethyl-aniline-ethyl carbonate; and methyl benzoate-ethyl carbonate.

As a definite relation exists between the degree of wetting (adhesion tension) of a solid by a solution and adsorption from that solution, investigations could be carried out on compounds of fractions of crude oils, and valuable data obtained as to the behaviour of displacement of oil by aqueous solutions.

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[6] A Method for the Measurement of Interfacial Tension of Liquid-Liquid Systems. Bartell and Miller. *J. Amer. Chem. Soc.*, 1928, **50**, 1961–1967.

A direct microscopic method has also been developed for the adhesion tension of liquids against transparent solids,[7] and the two methods are stated to be comparable.

Project No. 33 deals with *The Effect of Natural Gas upon the Viscosity, Surface Tension, Adhesion and General Extractability of Petroleums of Various Types.* It was concluded that the rate of flow of crude oil through unconsolidated sands is inversely proportional to the length of the flow tube, and that an unsaturated oil flows faster than one saturated with air or gas. The pressure-gradient for the entire length of a flow-tube is a straight-line curve for unsaturated oils. Viscosity is the prime factor affecting flow of unsaturated oils. Gas-saturated low-viscosity crudes respond to back-pressure better than air-saturated or unsaturated oils. Gas or air bubbles in a saturated mixture cause least resistance to flow when high back-pressures are used.

The Fundamentals of the Retention of Oil by Sand is the main subject of Projects Nos. 34 to 37. The distribution law for colloids in cells more than 5 mm. high was studied, and it was found that the Le Place-Perrin Law holds for the cell used, 0·83 cm. in height.

A great amount of time was devoted to investigating Antinof's claim to having developed a method for the accurate measurement of the surface tension of a solid against a liquid. It was concluded that this method is not reliable or applicable.

The measurement of the mobility of particles suspended in liquids was carried out by a new method, and the following data determined. Particles of 0·00015 cm. diameter or over have constant mobilities, while those of 0·0001 cm. diameter or less have orbits which fluctuate with time. The mobility of the large particles is constant with changes in the field strength and is slightly greater than that given by the A.C. method.

The Thermal Properties of Petroleum Hydrocarbons have been investigated under Project No. 38 and the results published.[8]

Project No. 39 deals with *Analysis of Crude Oil, its Distillates and Residues for Unsaturated Hydrocarbons and Surface-Active Matter by Means of the Dropping Mercury Cathode Method.* It was necessary to use a solvent with petroleum in order to allow the electrolytic current to flow. The removal of atmospheric oxygen must be carried out by the addition of about 30 mg. of sodium sulphite to the 25 c.c. of the aqueous solution to be electrolysed. The hydrogen ions for the reduction of hydrocarbons are best supplied by a

[7] Degree of Wetting of Silica by Crude Petroleum Oils. Bartell and Miller *Ind. Eng. Chem.*, 1928, **20**, 738–742; *Oil & Gas J.*, July 19, 1928, 102.

[8] Thermal Properties of Petroleum Hydrocarbons. Cragoe. *Bur. Standards Misc. Publ.*, No. 97.

buffer solution containing ammonium chloride besides the sodium sulphite.

Typical polarograms show two reducible substances in petroleum at about 1·0 and 1·4 volts, and experiments are being made to identify them.

Chemistry.

The Action of Oxygen (and Air) on Cyclopentane and Cyclohexane Hydrocarbons with Side Chains has been studied under Project No. 2 at temperatures below the normal boiling-points and at less than atmospheric pressures. The results so far obtained show a preponderant role assumed by the tertiary groups CH. To these the oxygen is attached. The peroxide formed evolves, and this evolution may maintain or rupture the cycle.

The investigation of *The Effect of Electrical Discharge upon Gaseous Hydrocarbons*, Project No. 8, confirms the theory that the liquid obtained by ionisation would be a mixture of hydrocarbons chiefly of higher molecular weights than the initial gas, but containing in solution some of the lighter gases also.

Under Project No. 15, *A Study of the Cyclopentane Fraction of Southern California Petroleum*, certain cyclopentane derivatives were prepared and the results made available.[9]

The aim of *A Study of Reactive Hydrocarbons*, under Project No. 16, was to obtain quantitative information regarding the energetics of breaking the carbon linkage in hydrocarbons. The values obtained give the equilibrium constant. The value in toluene solution at 24° C. is $0{\cdot}00242 \pm 0{\cdot}00011$.

Under the heading of *Non-Catalytic Thermal Decomposition of Pure Hydrocarbons and Related Compounds*, Project No. 18, the effect of high temperatures on propylene, allene, methyl-acetylene and diallyl has been studied and phenylbutylene, dicrotyl and dicyclohexyl have been prepared for similar studies.

For *A Study of the Relative Rates of Reaction of the Olefins*, Project No. 19, a special fractionating column has been developed in which samples from 1 litre to less than 10 c.c. can be successfully fractionated.

Measurements of the absorption rates of olefins by sulphuric acid have enabled factors to be calculated for the average rate per unit area of acid surface. While the rates for ethylene and propene are about 150 times smaller in 80 per cent. than in concentrated acid, their ratios remain at about 1 to 300 over this range. Butene dissolved three times as fast as propene in concentrated acid, but in dilute acid it was hard to tell which was greater.

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[9] Some Cyclopentane Derivatives. Case and Reid. *J. Amer. Chem. Soc.*, 1928, **50**, 3062–3065.

The belief that the normal butenes can be separated from propene by sulphuric acid absorption was proved to be wrong, and it was found that the effectiveness of 87 per cent. acid for separating propene from ethylene is decreased about 200 times by saturating with silver and nickel sulphates.

The work so far carried out on the *Isolation and Investigation of Nitrogen Compounds Present in Petroleum*, Project No. 20, has established that the structure of the $C_{12}H_{13}N$ base, boiling at 280·6° C., is 2, 3, 8-trimethyl quinoline, and its synthesis has been effected. Proof of structure and synthesis of the other $C_{12}H_{13}N$ base will be attempted. The $C_{14}H_{21}N$ compound is a hydro-aromatic heterocyclic compound that presents many structural possibilities.

Work is in progress under Project No. 21, *The Determination of Physical and Chemical Properties of All Types of Sulphur Present in Petroleum*, and in Project No. 28, *A Study of the Reactions of a Number of Selected Organic Sulphur Compounds*, the reactions of dilute mercaptan solutions with "doctor" solution have been studied. The amounts of lead mercaptides formed were found to depend on the nature of the solutions used. The synthesis of a number of the higher mercaptans has been undertaken and their physical properties determined.

The Metallic Constituents of Crude Petroleum have been investigated under Project No. 30, and the only generalisation at present apparent is that Wyoming, Mid-Continent and Pennsylvania oils are high in iron and alumina, but contain only traces of vanadium and nickel, while the oils from California, Texas and Oklahoma are low in iron and alumina but contain marked quantities of vanadium and nickel.

Oil Engines.*

By L. J. Le Mesurier, M.I.N.A. (Member).

The chief developments to record in oil engine construction during the past eighteen months are as follows :—

1. A general tendency has been evident in the direction of airless injection in all types of heavy oil engines. This applies not only to small engines of the 2-cycle and 4-cycle types, where air injection has now been practically superseded in engines up to about 50 B.H.P. per cylinder, but also in engines of the highest power produced for marine and land installations.
2. The low-compression hot bulb engine has been further superseded by medium or high-compression " cold start " engines, and the great majority of makers are now offering engines which do not require lamps, etc., for starting or running. In fact, adventitious aids to starting—such as chemical cartridges or electric ignition devices—are being eliminated ; and cold starting on direct injection of the fuel is provided for in most modern engines.
3. The double-acting principle has made rapid headway in engines of the larger sizes, and many examples of this type of engine have been put into service during the past twelve months.
4. Supercharging has been adopted by a number of well-known 4-cycle engine makers. The system, developed by Mr. Buchi, of the Swiss Loco. Works, was first applied to an engine built by the North-Eastern Marine and installed in the M.V. " Raby Castle," and the results were sufficiently encouraging to induce other makers to adopt the same or almost similar methods of supercharging. It is now claimed that the mean pressure in a 4-cycle engine can be augmented by as much as 40 per cent. without increase of heat stress and without appreciably adding to the loads on the running parts and bearings.
5. The majority of the leading oil engine makers have been giving close attention to the development of high-speed Diesel engines for traction, agricultural and marine purposes. The intention is to produce engines capable of competing with the petrol-paraffin type which still is the one chiefly employed for a class of work demanding engines of considerable power and low weight but running at more moderate speeds than

* Paper received Jan. 14, 1930.

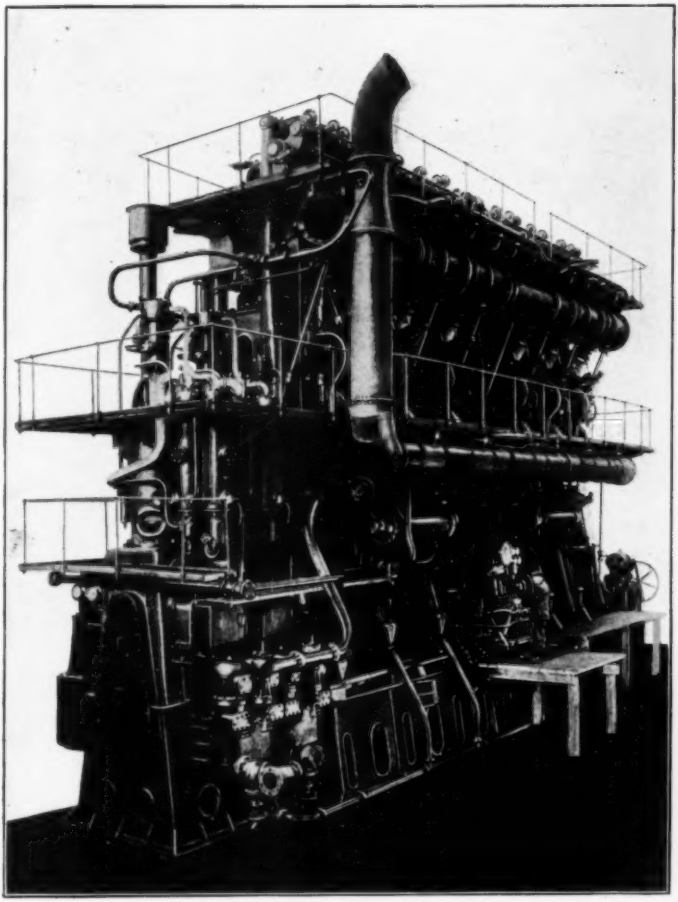
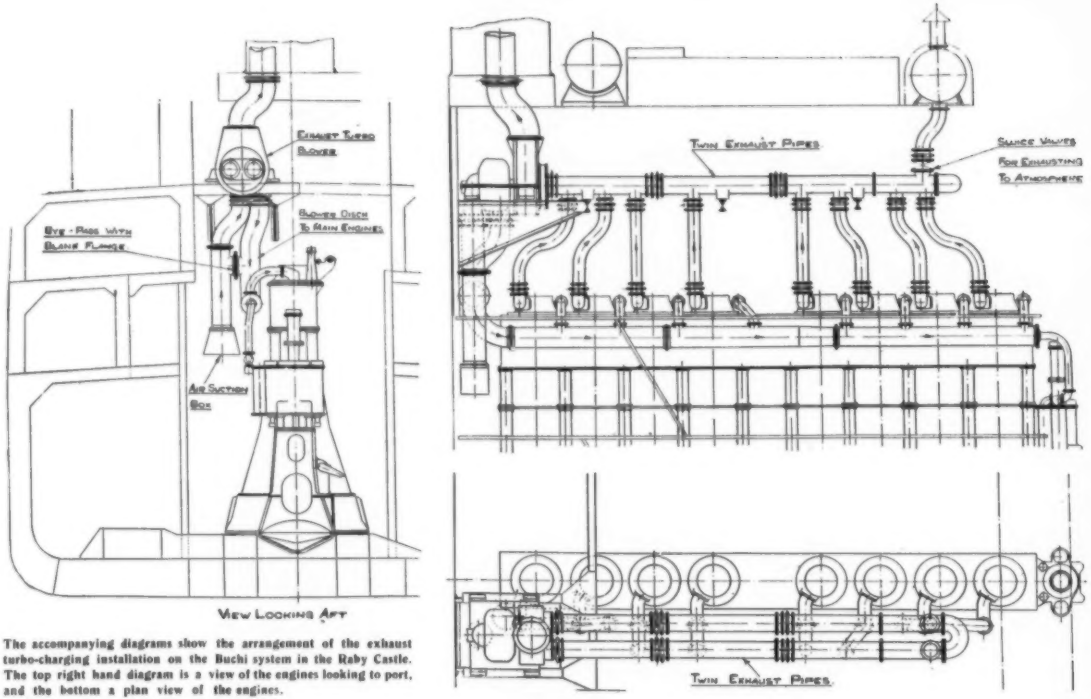


FIG. 1.

WERKSPOOR DOUBLE ACTING 4-CYCLE ENGINE. 3,400 B.H.P. 80 R.P.M.


Exhaust Turbo Blower
Bye - Pass with Blank Flange
Blower Discn To Main Engines
Air Suction Box
View Looking Aft
Twin Exhaust Pipes
Sluice Valves For Exhausting To Atmosphere
Twin Exhaust Pipes

The accompanying diagrams show the arrangement of the exhaust turbo-charging installation on the Buchi system in the Raby Castle. The top right hand diagram is a view of the engines looking to port, and the bottom a plan view of the engines.

FIG. 2.

NORTH-EASTERN MARINE ENGINE SHOWING EXHAUST AND SUPERCHARGING ARRANGEMENTS.

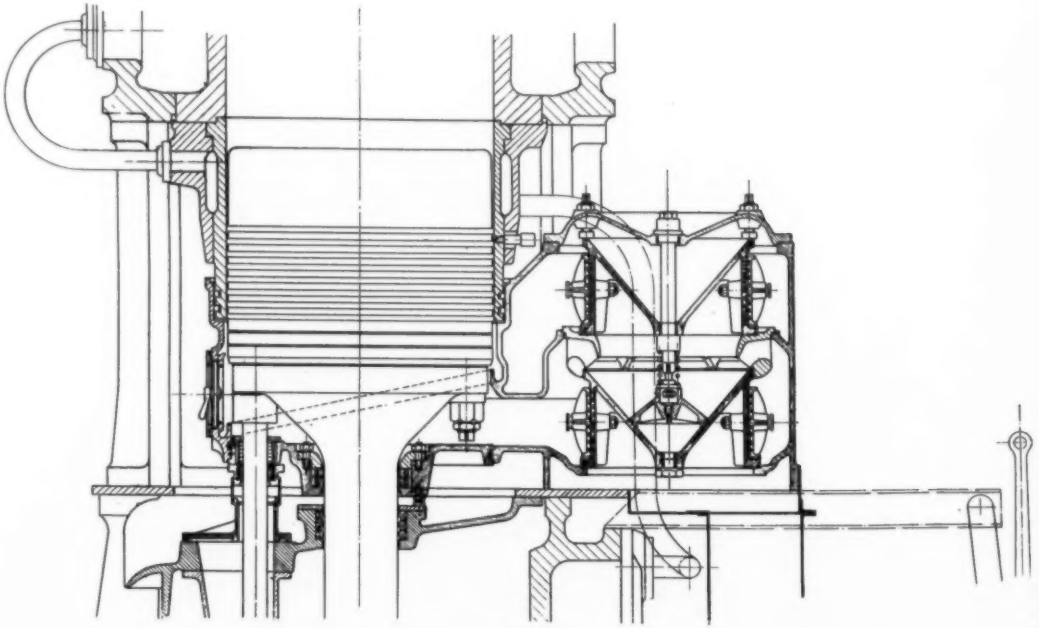


FIG. 3.

WERKSPOOR ENGINE. SUPERCHARGING ARRANGEMENT ON UNDERSIDE OF CYLINDER.

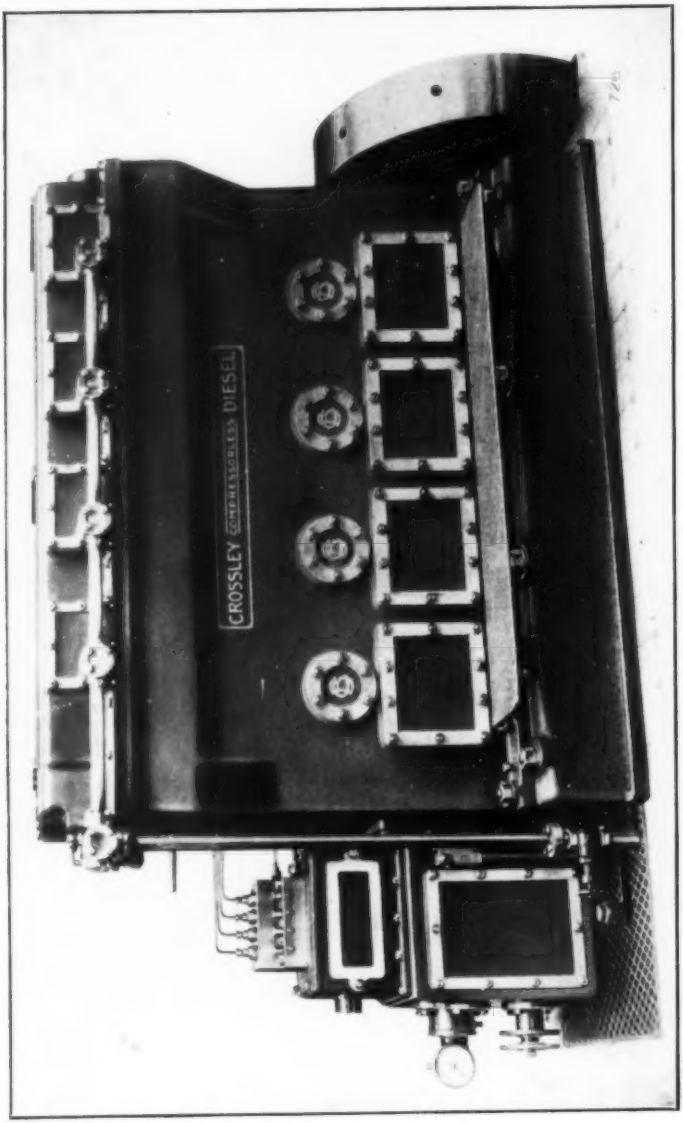


FIG. 4.

CROSSLEY AIRLESS INJECTION ENGINE.

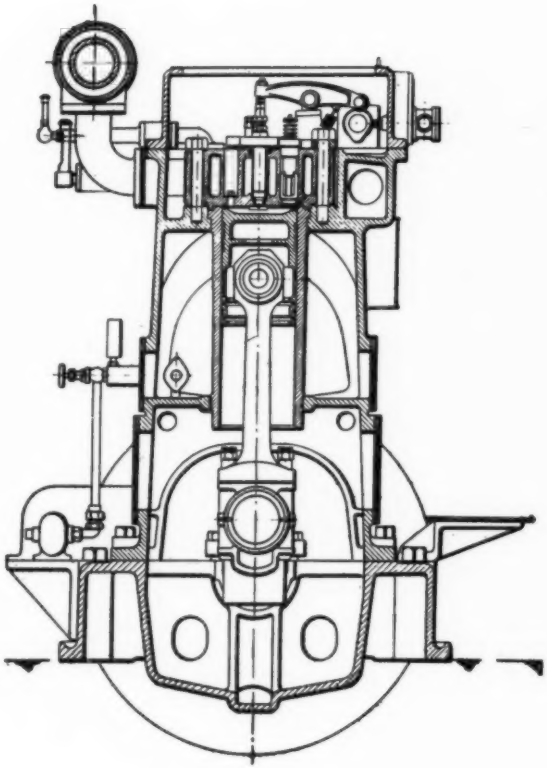


FIG. 5.

CROSSLEY AIRLESS INJECTION ENGINE.

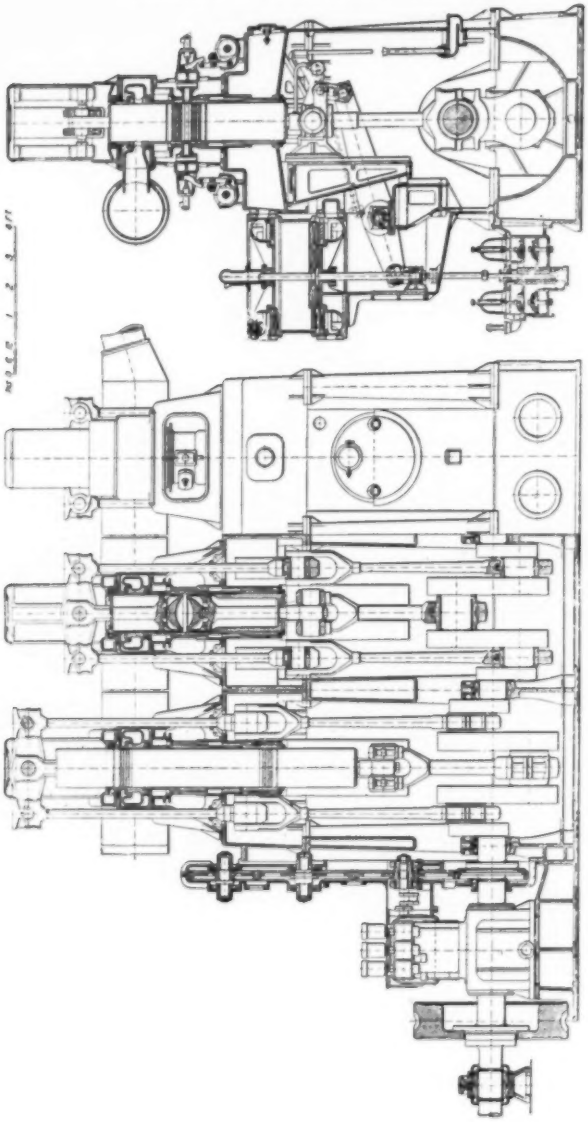


FIG. 6.

DOXFORD OPPOSED PISTON 2-CYCLE ENGINE.

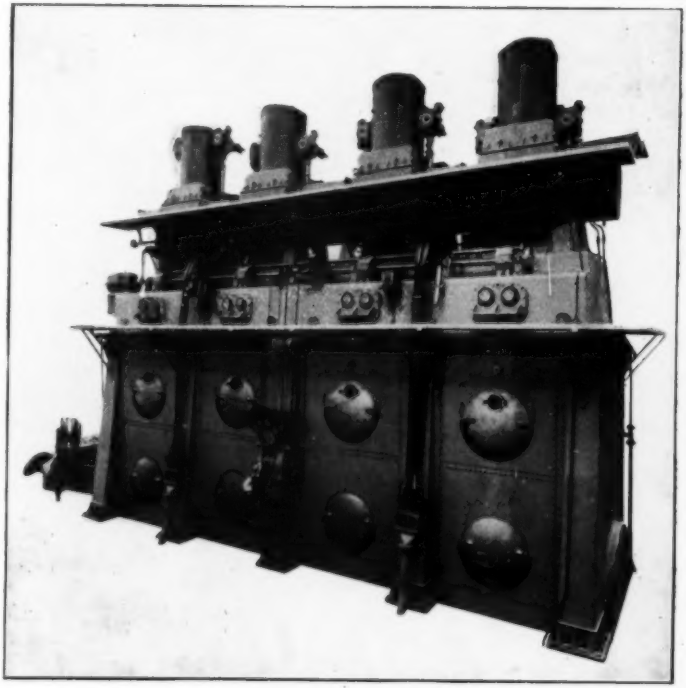


FIG. 7.

DOXFORD ENGINE. 3,000 B.H.P. 95 R.P.M.

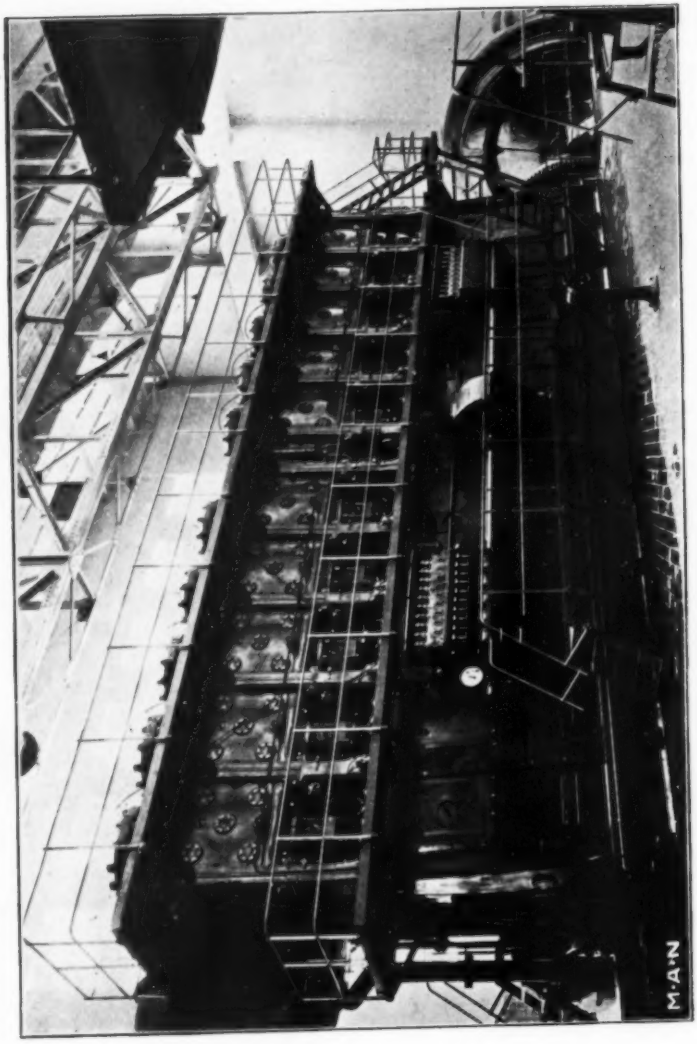


FIG. 8.

M.A.N. DOUBLE ACTING 2 CYCLE ENGINE. 11,700 B.H.P. 215 R.P.M.

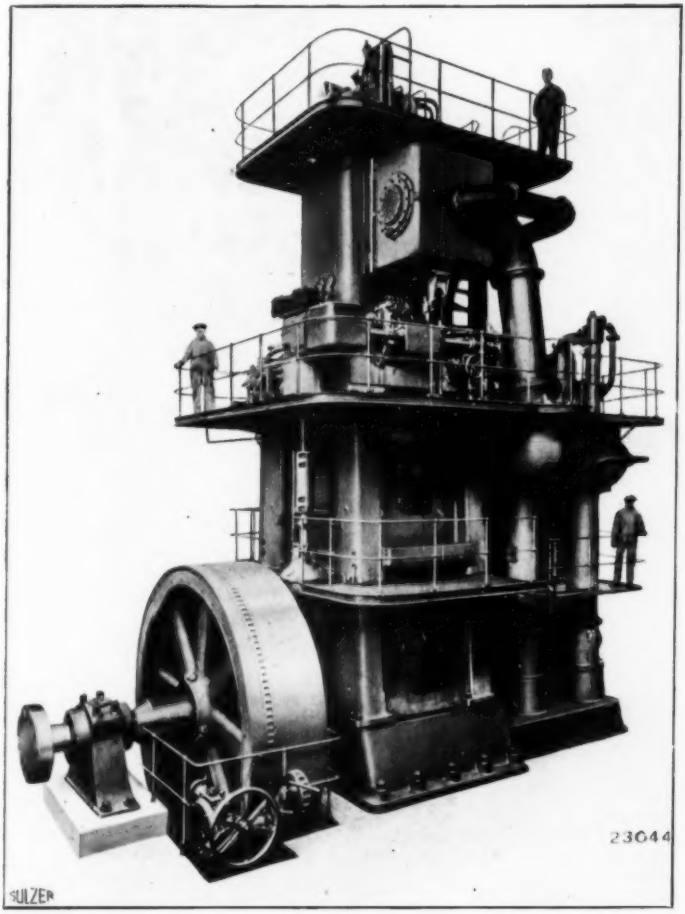


FIG. 9.

SULZER EXPERIMENTAL DOUBLE ACTING ENGINE. 2,000 B.H.P. 100 R.P.M.

M.A.N. DOUBLE ACTING 2 CYCLE ENGINE. 11,700 B.H.P. 215 R.P.M.

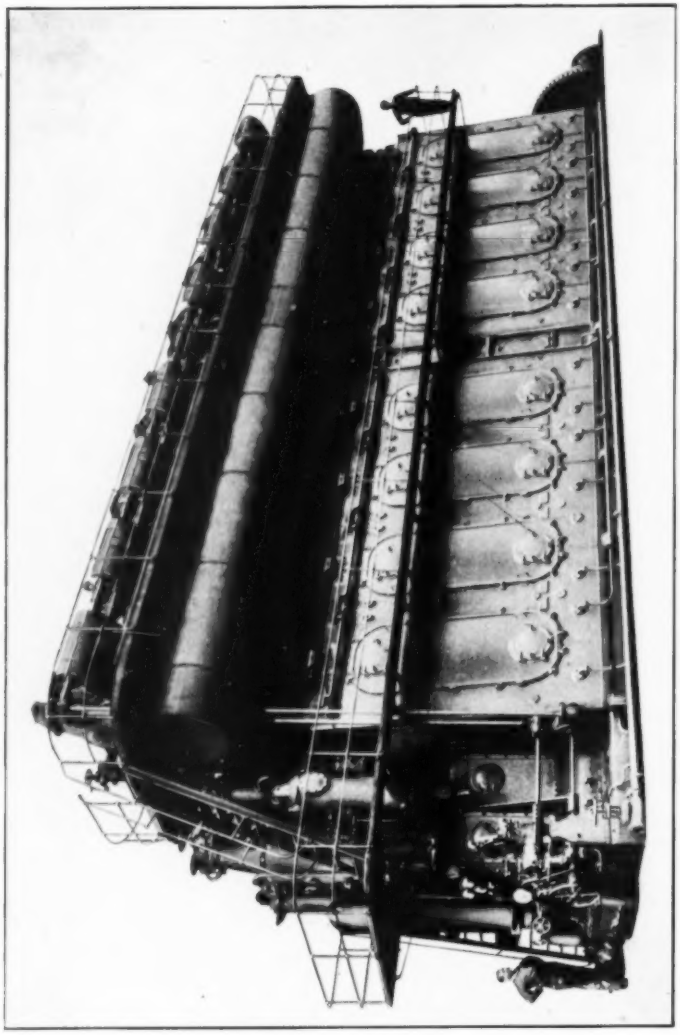


FIG. 10.

SULZER SINGLE ACTING 2-CYCLE ENGINE. 7,000 B.H.P. 100 R.P.M.

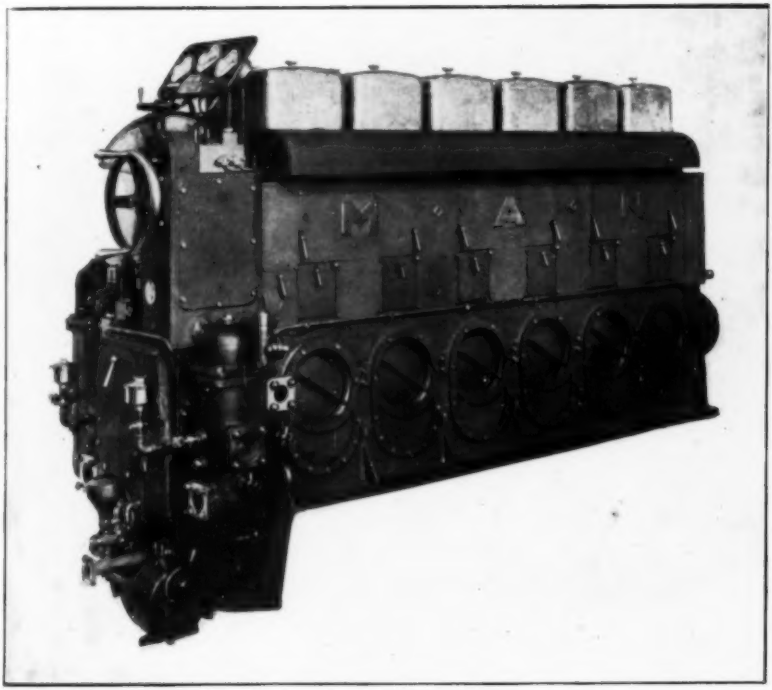


FIG. 11.

M.A.N. 4-CYCLE ENGINE. 300 B.H.P. 700 R.P.M.

SULZER SINGLE ACTING 2-CYCLE ENGINE. 7,000 B.H.P. 100 R.P.M.

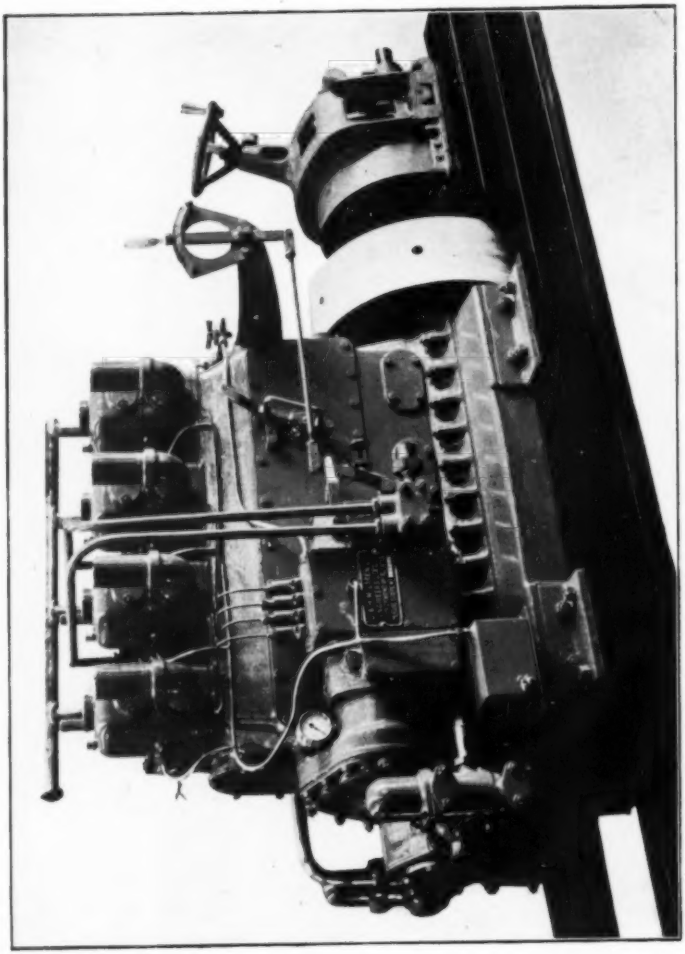


FIG. 12.

MCLAREN BENZ 4-CYCLE ENGINE. 60 B.H.P. 800 R.P.M.

FIG. 12.

MCLAREN BENZ 4-CYCLE ENGINE. 60 B.H.P. 800 R.P.M.

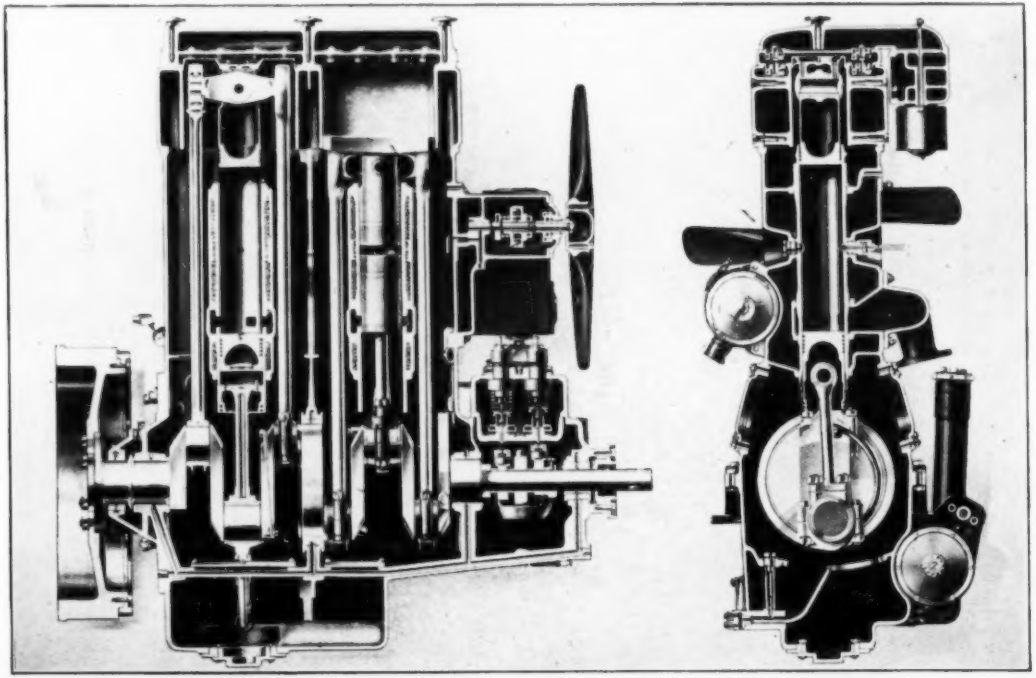


FIG. 13.

JUNKERS OPPOSED PISTON HIGH SPEED ENGINE.

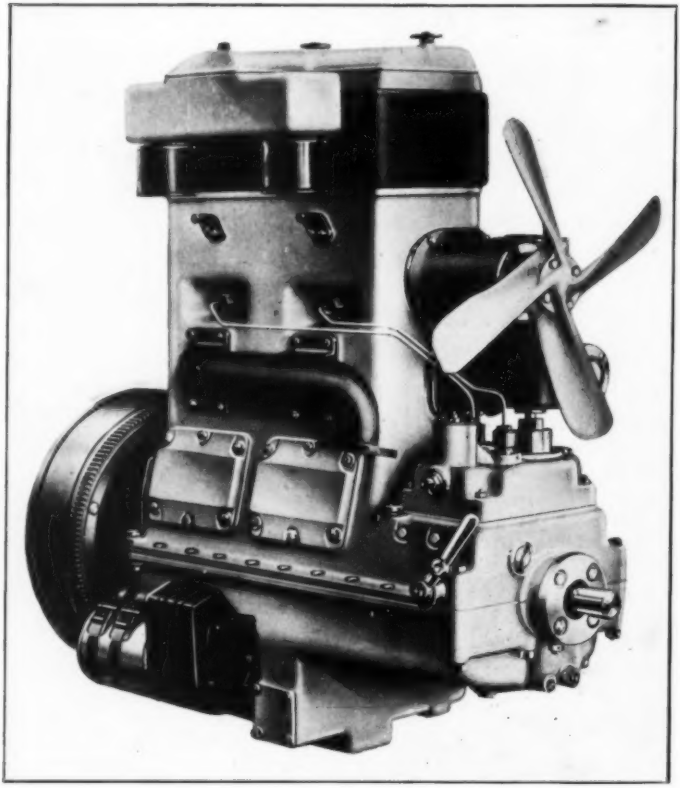


FIG. 14.

JUNKERS OPPOSED PISTON HIGH SPEED ENGINE

40 B.H.P. 1,000 R.P.M.

the ordinary petrol engine as fitted in motor cars. The demand for economy in fuel costs of the former type of engine led, in the first place, to the use of paraffin; and now the development is being carried a stage further, and it is only a question of time before engines using gas oil or Diesel grade fuels will be widely adopted for the purposes mentioned.

6. There is less tendency nowadays for adherents of either 2-cycle or 4-cycle types of engine to claim particular and exclusive advantages for the respective types since it is becoming more abundantly clear that each has its own sphere of usefulness. As a result, the majority of large oil engine manufacturers are now making both types of engine. Broadly speaking, the 2-cycle single-acting and double-acting types seem to be making definite headway where engines of large powers are required, while the single-acting 4-cycle type is chiefly in view for moderate and small powers. The double-acting 4-cycle engine, however, is still being built in considerable numbers, and must still be considered as an alternative to the 2-cycle type even for powers up to 10,000 B.H.P. In addition to this broad division, the 2-cycle type is also favoured in the case of engines of small power where low first cost of the engine is a chief consideration. Several examples are available of ships fitted with 2-cycle main propelling engines and 4-cycle auxiliary engines, illustrating the choice of type to suit a particular service. In the case of high-speed engines, development is chiefly concerned with the 4-cycle type, although there are a few promising designs embodying the 2-cycle principle.

Double-Acting 4-Cycle Engines.

There have been no particular developments of interest in connection with this type of engine, which is built in the United Kingdom by Messrs. Harland & Wolff and Kincaids, generally in accordance with Burmeister and Wain's design, and by the North-Eastern Marine and Hawthorn Leslies on the lines of the Werkspoor engine (Fig. 1). These engines are now in service in a large number of passenger and cargo ships, and appear to be giving good results, judging by the number of repeat orders which have been obtained. A number of single-screw tankers with Werkspoor double-acting engines were built for the Anglo-Saxon Petroleum Co., and satisfactory experience has now been obtained with the Lugt Zulver valve in service. This valve comprises a semi-rotary director valve which allows the cylinder to be connected either to the exhaust or induction pipe according to the position of the valve. The timing and control of the suction and exhaust strokes is effected by a

group of six mushroom valves in the cylinder head, which, in conjunction with the director valve, are used alternately for inlet air and exhaust.

The engines for the new White Star liner "Britannic" are to be of the double-acting Harland & Wolff 4-cycle type, each of 10,000 B.H.P., running at 110 r.p.m., the power being developed in ten cylinders of 840 mm. × 1500 m.m. dimensions. Separately driven air compressors will be provided for injection purposes. This installation when completed will comprise the largest 4-cycle double-acting engines in existence.

Single-Acting 4-Cycle Engines.

Considerable progress has been made in the application of supercharging to 4-cycle single-acting engines. North Eastern Marine, Burmeister and Wain and Werkspoor have all used a system in which an exhaust-driven turbine is direct coupled to an air turbo blower. The charging pressure is about 4 lb. per sq. in., and by arranging an overlap on the timing of the inlet and exhaust valves, and also by suitably designing the exhaust system, it is possible to scavenge completely the clearance space at the top of the cylinder during the period while inlet and exhaust valves are open. This is an essential feature of the supercharged engine, and it is claimed that approximately double the weight of air can be supplied as compared with a normal engine, so that even with 40 per cent. more power the mean temperature of the cycle is no greater than in a normal engine (Fig. 2).

Messrs. Werkspoor, in addition to applying the exhaust-driven blower system, have also made an engine in which the underside of the cylinder in a single-acting 4-cycle engine is made to act as a scavenge pump (Fig. 3). The pump is fitted with automatic valves so that a charge of air is delivered every revolution, thus providing 2-cylinder volumes, less allowance for volumetric efficiency and piston rod volume, of air to the working end of the cylinder. While this system reduces the efficiency of the engine on account of the power absorbed by the scavenge pump, it has the advantage of simplicity; and, furthermore, the exhaust gases can be made use of in a waste heat boiler, so that the overall efficiency of the plant is not appreciably affected. Amongst builders of smaller engines, Davey Paxmans have used a rotary blower driven from the crankshaft for supercharging.

Airless injection is being used in an increasing number of 4-cycle engines of large power. This system, although first used in the Vickers crosshead engine, was chiefly confined to engines of the trunk piston type, but recently Harland and Wolff and Burmeister

and Wain have installed trunk piston engines of this kind in cross-Channel boats. The Harland and Wolff engines have 10 cylinders of 630 mm. × 980 mm. stroke and develop 3000 B.H.P. at 160 r.p.m. The fuel injection system includes separate pumps for each cylinder with sleeve valve control and an automatic spring-loaded fuel injection valve. The design also provides for a slight degree of supercharging by means of an electrically-driven turbo blower.

Burmeister and Wain have recently adopted both airless injection and supercharging in their standard slow-running crosshead engines, a notable example being the twin-screw machinery fitted in the M.V. "Agamemnon" of the Blue Funnel Line. The engines have each 8 cylinders, of 740 mm. × 1500 mm. dimensions, and would normally develop about 3000 B.H.P. at 110 r.p.m. With supercharging, however, the normal output has been increased to 4300 B.H.P. with a maximum overload up to 5090 B.H.P.

The M. A. N. Co. are building large airless injection 4-cycle engines. A particular example is an engine of the crosshead type with 9 cylinders of 720 mm. × 1400 mm. stroke to develop 3600 B.H.P.

Sulzers continue to use the 2-cycle type exclusively for propelling machinery, but they also manufacture marine auxiliary engines, and large numbers of stationary engines of the 4-cycle type. Within the past year they have installed quite a number of 4-cycle engines as marine auxiliaries and have recently completed engines of a new airless injection type for stationary work. An engine of this type to develop 1000 B.H.P. at 215 r.p.m. has 6 cylinders with dimensions 480 mm. × 700 mm.

A new type of engine running at moderately high speeds has been developed by Vickers-Armstrong. The design is intended to apply chiefly to Naval craft where a high power/weight ratio is essential. The design follows generally the lines of the Vickers submarine type used during the war and includes cam-controlled fuel valves and independent fuel pumps which can be connected to a common rail. The weight of the engine comes out at about 40 lb. per B.H.P.

In the smaller sizes of 4-cycle engines used for stationary purposes and ship auxiliaries, airless injection is being strongly favoured. There are, however, certain cases, particularly for marine auxiliaries, where the air compressor is advantageous, as, by fitting an oversize blast air compressor, surplus air in available for charging the air-starting reservoirs, thereby doing away with the necessity for a separate auxiliary compressor.

An engine of quite unorthodox design has been produced by Peter Brotherhood. This engine, which has been developed in conjunction with Ricardo, is fitted with an aluminium piston working in a hardened and ground steel liner which is driven by a rocking arm and acts as a sleeve valve for regulating the inlet and exhaust.

The engine has been built as a 6-cylinder unit developing 50 B.H.P. per cylinder at 900 r.p.m., and the weight is said to be about 40 lb. per B.H.P.

Amongst builders of cold start 4-cycle horizontal and vertical engines, Ruston Hornsby, National Gas Engine Co., Blackstones and Gardners continue to supply increasing numbers of their well-known types. The behaviour of these engines in service has been such that only improvements in minor detail have been made in the later types. The importance of having such engines able to run successfully on the widest possible range of fuels is being appreciated, and in some cases the fuel injection system has been modified to secure a greater degree of flexibility in this respect.

Crossleys have introduced new designs of vertical and horizontal engines with chain-driven cam shafts, a feature which seems to be gaining in popularity. The horizontal engine is of the enclosed type, and has inlet and exhaust valves placed parallel with the centre line of the cylinder, a practice common in vertical engines, but which, so far, has not been used to any great extent in horizontal engines (Figs. 4 and 5).

There is, on the whole, a decided tendency to substitute high-compression cold start engines for the low-compression hot bulb engines, and several makers of 4-cycle engines have practically abandoned the latter type. The cheaper cost of production of the low-compression engine is, however, a factor which may give further life to this type of engine.

Double-acting 2-cycle Engines.

In this class the Doxford opposed-piston airless injection engine can claim to have by far the longest experience, and it is a tribute to the original design that the latest types differ only in minor points of detail from the original design (Figs. 6 and 7).

The chief improvement made in the new engines is the complete balancing of primary and secondary forces obtained by making the stroke of the upper piston slightly less than that of the lower piston, and adjusting the masses and disposition of the cranks to give a complete balance.

Lever-driven scavenge pumps have also recently been introduced, enabling the overall length of the engine to be reduced.

The M.A.N. Co., who in 1927 built the four engines for the M.V. "Augustus," developing a total of about 25,000 B.H.P., have since put into service a large number of double-acting engines, the total output of this type delivered, or under construction for merchant vessels only, being approximately 250,000 B.H.P. Airless injection has been adopted in some of the later engines, and Fig. 8 shows a 10-cylinder M.A.N. engine of 11,700 B.H.P. at

215 r.p.m. which forms part of a 15,000 K.W. peak load equipment at Hennigsdorf sub-station of the Berlin electricity works. On trials this engine was stated to have a fuel consumption of only 0.36 lb. per B.H.P./Hr. exclusive of scavenge blower and auxiliaries, or about 0.385 lb. with auxiliaries included. Incidentally, the use of oil engines for dealing with peak loads and auxiliaries in coal-fired power stations has definite advantages, which are becoming more generally appreciated, and considerable developments in this direction may be anticipated.

The first Richardson Westgarth double-acting engine, a 3-cylinder engine of 1200 B.H.P. at 85 r.p.m., has been put in service in an oil tanker with promising results. This engine can be regarded as the first purely British design of double-acting engine employing a single piston with piston rod and crosshead. There are several very interesting points in the design, which has been developed with special regard to accessibility to the cylinders and working parts, a feature which is particularly necessary in marine work. The system of airless injection adopted includes separate fuel pumps for each cylinder, and fuel control on the suction valves for regulating the quantity of fuel and—by means of a spill valve—for determining the end of the injection period. The cylinder covers are so fitted that the liner is free to expand endways, and the scavenge is effected by a double-acting pump geared to run at a higher speed than the crank shaft.

Deutsche Werke have also built a new double-acting type of engine which has been installed in a fast twin-screw cargo ship for the Hamburg South American Line. The engines have air injection, and separately driven turbo blowers for scavenging. With 6 cylinders of 650 × 1050 mm. dimensions, each engine is designed to develop 4200 B.H.P. at 115 r.p.m.

Sulzers have now in hand a double-acting engine of 8 cylinders, with dimensions 700 × 1200 mm., designed to develop about 7000 B.H.P. at 106 r.p.m. The design is based on a 2000 B.H.P. single-cylinder experimental engine which has been put through exhaustive shop tests during the past two years (Fig. 9).

One of the most interesting double-acting developments is the engine just completed by Burmeister and Wain for the East Asiatic Co. Burmeister and Wain have been for many years the principal exponents of the 4-cycle type and, in now building a double-acting 2-cycle engine, they evidently foresee the possible advantages of this type for a particular kind of work.

The first engine of this kind, constructed for the East Asiatic Co., is designed to develop 7200 B.H.P. at 100 r.p.m. in 6 cylinders of 620 × 1400 mm. dimensions. The brake mean pressure is 90 lb. per sq. in., or more than 50 per cent. higher than in other

engines of this type with piston rods, or about the same mean pressure as in the opposed-piston type. The design includes piston-controlled exhaust valves, which are placed in the upper and lower cylinder covers, the latter valve being in the form of an annulus surrounding the piston rod. The scavenge is effected through a row of ports in the centre of the cylinder so that the combustion products are swept outwards from the centre of the cylinder through the exhaust piston valves in the covers. A curious feature of this engine is that the valves themselves acts as pistons which return power to the crankshaft through the valve-operating mechanism and, in fact, the design can be regarded as of the opposed-piston type with the valves at each end of the cylinder acting as small power pistons.

Single-Acting 2-Cycle Engines.

The largest single-acting 2-cycle cylinder so far built is fitted in the Brown Sulzer engine in the twin-screw M.V. "Rangitiki" belonging to the New Zealand Shipping Co. These engines have 5 cylinders of 900×1600 mm. and develop nearly 900 B.H.P. per cylinder, or 4,500 B.H.P. per engine. This indicates the possible developments of high power with the single-acting 2-cycle type. Another interesting 2-cycle Sulzer engine is an 8 cylinder unit of 7040 B.H.P. at 100 r.p.m. Built for a single-screw motor-ship belonging to the Netherland S.S. Co. (Fig. 10), the cylinder dimensions are 820×1440 mm., which may be compared with the double-acting 2-cycle Sulzer type mentioned above.

In the small sizes of 2-cycle engines the development is all in the direction of cold start airless injection. Petters, Gardners and many other builders of the 2-cycle hot bulb crankcase-scavenge engine have introduced new types with high compression and cold start, the general features of the hot bulb type being otherwise maintained.

The brake mean pressure in this type of engine is still round about 40 lbs. per sq. in. and consequently, although the engine is extremely simple, the cylinder dimensions for the same output are practically the same as in the 4-cycle engine which usually operates at nearly double this mean pressure.

High-Speed Engines.

Small high-speed engines, running at 1,000 r.p.m. and developing from about 10 to 20 B.H.P. per cylinder, are being developed by a large number of firms. The designs generally follow motor car practice and comprise 2, 4 and 6 cylinder units. The system of fuel injection, employing a prechamber, is adopted in the Benz, McLaren Benz, and Deutz engines, while direct injection is used

in the M.A.N. type, all these being 4-cycle engines. Saurers employ an offset combustion chamber in which a high degree of turbulence is obtained, the fuel being injected at the throat connecting the combustion chamber with the cylinder. The weight of engines of this type amounts to about 20 to 30 lbs. per B.H.P. (Figs. 11 & 12).

The accurate control of fuel in the minute quantities required in these types of engines demands the highest quality of workmanship in the fuel pumps and fuel injection valves and has led to the development of special pumps and fittings which are manufactured as accessories, much in the same way as the magneto is manufactured for the ordinary petrol engine. In particular the Bosch Co. make a pump and fuel injector which is fitted to a number of different types of engine. No doubt other types of pumps will be similarly developed as specialised fittings to deal with this class of work.

The Hesselman engine is another interesting attempt to provide a high-speed heavy oil engine without the necessity of high compression. The compression is only slightly above that of the normal petrol engine and the fuel is injected slightly before the top of the stroke and vaporised by the heat of an uncooled lip which is provided either on the piston or cylinder head. The actual ignition of the fuel is effected by a sparking plug which is shielded from the direct impingement of the fuel. The engine requires to be started on petrol and is turned over to heavy fuel when sufficient heat is available for vaporisation. The trials so far carried out have been made on fuels corresponding to gas oil, but it is claimed that Diesel grade fuel can also be used satisfactorily.

The 2-cycle engine with opposed piston has been developed in the small sizes by Junkers. A large number of moderate speed stationary engines of this type are already in service and good progress has been made with the design and manufacture of higher speed types suitable for traction and marine purposes (Figs. 13 & 14).

The tendency is noticeable towards higher speeds, multi-cylinder, and airless injection, for all engines of moderate or small powers, and all these types of engine require a superior grade of fuel than has hitherto been found necessary for large slow running engines.

Up to the present the fuel requirements are so small that it is possible to select suitable fuels from the wide range of Diesel fuels available. It seems necessary, however, in order to reap the full benefit of the economy in fuel costs of the high-speed Diesel engine, that every effort should be made to develop the engine in such a way that all grades of Diesel fuel can be used. Otherwise, if there is a large demand for special fuels, such as gas oil, it will inevitably mean higher fuel costs for engines requiring such fuel and, consequently, a preference on behalf of buyers for engines capable of using a wider range of fuels.

Oil and Gas Developments in Western Canada During 1929.*

By G. S. Hume.

The most extensive programme of drilling for oil and gas in the history of western Canada was undertaken during 1929. The drilling activity may be summarized as follows:—

Turner Valley—Ninety wells drilling or completed during the year.

Foothills outside of Turner Valley—Thirty-three wells drilling. Several wells abandoned either on account of drilling difficulties or unfavourable prospects. No successful completions to date but conditions regarded as highly favourable in certain areas.

Plains of Alberta—Thirty-five wells drilling. Several successful oil and gas wells completed. Two prospective oil fields—Red Coulee and Ribstone—and one gas field—Kinsella—discovered.

Saskatchewan—Eight wells drilling. No successful completions to date.

Manitoba—One well drilling. No completions during year.

The peak of production[1] for the year 1929, for Turner Valley was reached in October when 102,466 barrels of naphtha and crude oil were delivered to the Imperial and Regal refineries in Calgary. This represents an average daily production of 3,305 barrels, compared with 1,027 barrels per day for the same period in 1928, or 1,562 barrels per day for December, 1928, the highest record for that year.

There are several oil and gas horizons in Turner Valley. Light crude oil is produced from Lower Cretaceous and Jurassic horizons and naphtha with gas from the underlying Palæozoic limestone. During 1929, several wells secured commercial production of oil from the Cretaceous and Jurassic horizons and the following wells were completed to the limestone horizon and secured a flow of gas and naphtha.

East Crest No. 1; Foothills No. 2; Home Nos. 1, 2 and 3; Mayland No. 1; McLeod Nos. 3 and 4; New McDougal-Segur No. 2; North-West Associated No. 1; Royalite Nos. 13, 14, 17, 18 and 19; Regent No. 1; Spooner No. 2. Besides these a number of wells are drilling in the limestone and production may be expected at an early date. The most spectacular developments during the year 1929

* Paper received January 13, 1930, and published by permission of the Director, Geological Survey of Canada.

[1] *Bull. Can. Inst. Min. and Met.*, December, 1929, p. 1322. (December production figures not available at time of writing.)

centred around the south central part of Turner Valley. Home No. 1 well came into production on February 17th, at a depth of 5,280 feet with a naphtha flow of about 600 barrels per day. Since that time Home Nos. 2 and 3 and Northwest Associated No. 1 have been added to the producers of this area, all of these wells have a capacity for large production but a certain amount of difficulty has been experienced in keeping the wells on steady production. This difficulty is common to many of the naphtha wells in Turner Valley and has been assumed to be due to the low temperature produced in the wells by the rapid expansion of the gas. During the autumn of 1929 Royalite No. 4 well, for the first time since the well was completed in 1924, ceased producing for a time. It has since come back "into partial and later into almost the full former production but no deliveries were made to the refinery during October."[1]

Among the wells that have secured production of oil from horizons above the limestone is Merland No. 1, in the extreme south of Turner Valley on the north slope of Longview hill in section 27, township 18, range 2. This production was secured from the Jurassic and is significant because it greatly extends the producing area of Turner Valley to the south. A short distance south of the Merland well the Turner Valley anticline plunges rather steeply to the south and the anticlinal structure disappears, but there is still room for some more wells south of the Merland well.

In the north end of Turner Valley, Freehold No. 1 well in section 3, township 21, range 3 has secured some production and the well is being deepened to lower horizons. About 1½ miles north of the Freehold well is the Invaders well. It is understood that this latter well has secured some oil shows of non-commercial importance at an horizon not productive in Turner Valley and drilling is being continued. The Invaders well is the most northerly drilling site in Turner Valley and according to recent work carried out in this area by the Geological Survey there is a thrust fault of more than 2,000 feet throw a short distance north and east of the well. It is considered that for practical purposes this thrust fault marks the northern end of Turner Valley.

The approximate length of Turner Valley as thus defined is 17 to 18 miles, and throughout the whole distance the prospects for production are excellent. The width of Turner Valley is slightly more than one mile at the maximum.

Drilling has shown that the structure within the anticline, that composes Turney Valley field, is greatly complicated by faulting. Drilling depths to the limestone are difficult to predict, and during drilling operations unsuspected faults have been discovered where

[1] *Bull. Can. Inst. Min. and Met.*, December, 1929, p. 1320.

none was known. In a general way it has been found that the gas from wells highest on the structure has a lower naphtha content than that from deeper wells. The naphtha content shows considerable variation for different wells.

In the foothills outside of Turner Valley there has been considerable drilling activity. The work of the Geological Survey has shown that an anticline extends from the Highwood River area in ranges 2 and 3 to the Jumpingpound creek in range 5, a distance of more than 40 miles. This anticline, like that of Turner Valley, is flanked by Belly River strata. The east flank is a ridge composed of a series of hills, and can be traced with few breaks from the Highwood River northwest to Jumpingpound creek. The west flank is irregular and therefore the main anticlinal structure has a somewhat variable width up to 6 miles. This major anticlinal structure is itself folded into subsidiary anticlines and synclines and cut by many thrust faults. Blairmore strata, which are encountered at depths of 1,500 to 3,500 ft. in Turner Valley, are exposed over wide areas within this major anticline, and numerous gas and oil seepages occur in several localities. Not only is plunging of the main anticlinal structure a marked feature, but plunging of the minor anticline superimposed on it occurs. It is thought that many of the subsidiary anticlines superimposed on the main structure are favourable for oil and gas, and drilling is proceeding in a number of places. The Imperial Highwood No. 1 well was drilled on a subsidiary anticline on this main structure. After penetrating the Palæozoic limestone for some distance this well encountered complicated structural conditions and passed back into Jurassic shales. The well was abandoned, but another well has been started on the same structure about a half mile west of the first. Other wells drilling on subsidiary anticlines within the main anticlinal structure have encountered encouraging amounts of oil and gas, but none has been completed.

One of the disappointing developments of the year was the discovery of a low angle thrust fault underlying the so-called New Black Diamond anticline. This anticline lies about two miles west of Turner Valley on Sheep River, and east of the Highwood-Jumpingpound anticlinal structure. It has now been proven that the anticlinal structure which is apparent on the surface is but superficial, and two wells have encountered Belly River strata under a low angle thrust fault which at the surface thrusts the Benton shales over the Belly River rocks. This drilling has revealed that the low angle thrust fault cuts off the west limb of a syncline, so that further drilling on the New Black Diamond structure is probably not warranted by the prospects for oil and gas.

The Imperial Jumpingpound well drilling on an anticline on the

eastern edge of the Foothills south of Bow River has also encountered unfavourable structural conditions and been abandoned. The well passed from Jurassic to Cretaceous (Belly River) strata under a thrust fault. This is the second unsuccessful well in the Jumping-pound area, but it is understood another well may be drilled.

On the Plains there has been considerable drilling at Wainwright, and a number of new wells have been completed during the year. The oil from the Wainwright wells is about 18 to 20 degrees Baumé. Two small refineries extract distillate from the oil. The distillate is used locally in tractors, etc., and up to the present the demand has been exceeding the supply.

East of Wainwright in the Ribstone field, on the Saskatchewan boundary, Meridian No. 1 well has been successfully completed as an oil well with a capacity of 65 to 100 barrels of oil per day from a depth of 1,830 ft. Several thousand barrels of oil have already been pumped from the well, and the oil is being used as fuel in drilling three more wells.

In southern Alberta, in the Coutts area, close to the International Boundary, and north of the Kevin-Sunburst field of Montana, the Vanalta well has opened up a new prospective oil field in the Red Coulee district. The capacity of the well is reported[1] to be 60 barrels per day of 31 degree Baumé oil. This is considered one of the most important developments of the year, on account of the quality of the oil produced from a Plains structure. A few years ago the Imperial Oil Company's Red Coulee well was abandoned after getting oil in non-commercial amounts. It would seem, however, that the Vanalta well has proven the value of the structure, and several wells will be drilled at once.

In the Kinsella district about 8 miles east of Viking, in north central Alberta, the Kinsella-Duluth well on section 29, township 48 range 10 west of 4th meridian, has encountered a dry gas flow of 27 million cubic feet per day. This is a new gas field. The Geological Survey has no information regarding the details of the structure.

During the year a number of oil companies have had geological parties exploring for new prospective oil and gas structures in the foothills and plains. It is understood that several new structures have been discovered, and these are likely to be drilled during 1930.

[1] *Bull. Can. Inst. Min. and Met.*, December, 1929, p. 1321.

British West Indies.*

By A. P. Catherall, B.Sc., M.I.Min.E., A.M.Inst.M.M. (Member).

Trinidad

During the period under review there has been a steady increase in the development of the oil industry of the colony. Whilst the majority of the production is still obtained from the Fyzabad-Morne L'Enfer-La Brea districts intensive development has been undertaken in the Palo Seco district with the result that an increase in production of a high-grade oil has been obtained. Drilling has also been undertaken at Guayaguayare, but owing to the absence of transport facilities operations have been of no great magnitude. A pipe-line has now been laid to connect this field with the main line of the Trinidad Leaseholds, Ltd., at Barrackpore.

Both the footage drilled and quantity of oil produced during the period have shown a considerable increase as will be seen from the table which follows :—

	Year ended December 31, 1927.		Year ended December 31, 1928.		Nine months ended September 30, 1929.	
	Feet drilled.	Oil won. Brls. of 35 gals.	Feet drilled.	Oil won. Brls. of 35 gals.	Feet drilled.	Oil won. Brls. of 35 gals.
Crown Lands	119,284	2,362,150	130,630	4,141,816	142,483	3,439,565
Private Lands	121,440	3,018,314	114,010	3,542,634	75,870	3,100,904
	240,724	5,380,464	244,640	7,684,450	218,353	6,540,469

The accompanying diagram shows these figures over the period 1911-12 to 1928.

During the period January 1, 1928, to September 30, 1929, 275 new wells were drilled, of which 168 were on Crown lands and 107 on private lands, and in 95 of the wells oil was struck.

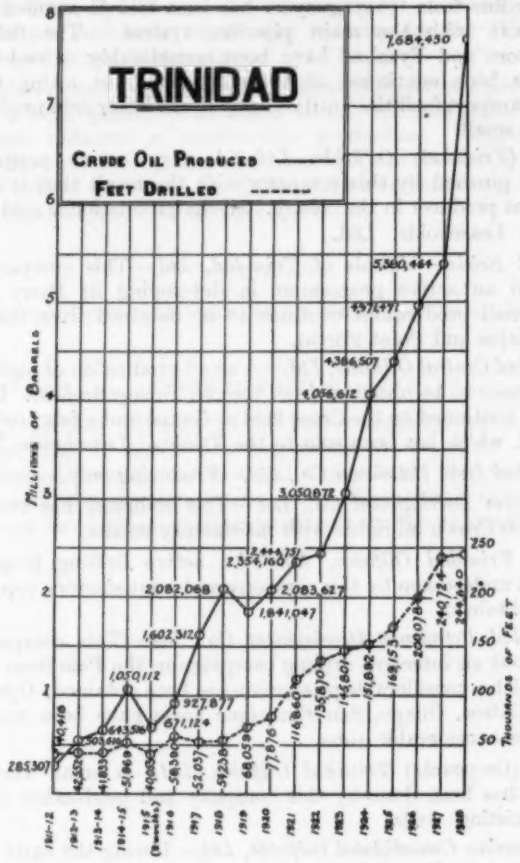
The total number of wells drilled in the colony to September 30, 1929, is 1,587.

The value of petroleum products exported has also increased, and in 1928 amounted to 37·3 per cent. of the total value of domestic products exported.

There has been an addition of one new shipping depot during the period under review, *viz.*, that of the Trinidad Oil Fields Operating Co., Ltd., at La Brea where a pier capable of taking ocean-going vessels for loading oil for export has been constructed. A refinery has been erected by the West India Oil Co. to deal with the crude oil produced by the Trinidad Oil Fields Operating Co., Ltd.

* Paper received January 3, 1930.

The Trinidad Leaseholds, Ltd., over the period, has had under construction considerable additions to its refining plant at Pointe-a-Pierre, including additional refining units, Edelenau Plant for the treatment of kerosine, etc.



The following is a brief summary of the operations of the oil companies working in the colony:—

Trinidad Leaseholds, Ltd.—This company has now fallen to the second place of the producing companies in the colony. It is,

however, by far the largest exporter of petroleum products, some 70,000 tons per month being treated in its refinery. All the production of the Apex (Trinidad) Oilfields, Ltd., and some other smaller companies has been pruchased. The old 6-in. pipe-line from its fields at Fyzabad has been replaced by a 10-in. line and a 6-in. pipe-line from Guayaguayare has been laid to connect up at Barrackpore with the main pipe-line system. The fields at Barrackpore and Fyzabad have been considerably extended and work has been continued at Guayaguayare, but owing to the lack of transport facilities until recently in the latter field production has been small.

Apex (Trinidad) Oilfields, Ltd.—A very active programme has been pursued by this company with the result that it is now the largest producer in the colony. All the production is sold to the Trinidad Leaseholds, Ltd.

United British Oilfields of Trinidad, Ltd.—This company has continued an active programme in developing its Parry Lands field. Small productions continue to be obtained from the fields at Los Bajos and Point Fortin.

Trinidad Central Oilfields, Ltd.—A small production of high-grade oil continues to be obtained from the old Tabaquite field. Drilling has been continued on the Cruse field at Guapo and a fair production obtained, which has been sold to the Trinidad Leaseholds, Ltd.

Trinidad Lake Petroleum Co., Ltd.—Producing only.

Petroleum Development Co., Ltd.—This company has continued drilling on Crown oil rights with satisfactory results.

Kern Trinidad Oilfields, Ltd.—An active drilling programme has been undertaken by this company and a satisfactory production of oil obtained.

Trinidad Petroleum Development Co., Ltd.—This company has carried out an intensive drilling campaign in the Palo Seco district from which a considerable production has been obtained. Operations in the Cedros, Guapo, San Francique areas have been suspended for some considerable time.

New (Oropouche) Trinidad Oilfields, Ltd.—A small amount of drilling has been done by this company and production obtained from existing wells.

Venezuelan Consolidated Oilfields, Ltd.—During the early part of the period under review work was confined to developing its properties in the Fyzabad district from which a fair production was obtained. Since the early part of this year there has been an active development programme on Crown oil rights in the Palo Seco district with a considerably increased production.

Palo Seco Oilfields (*C. C. Stollmeyer*).—Drilling continued during the period and a small production was obtained.

Trinidad Oil Fields Operating Co., Ltd.—This company has been acquired by the West India Oil Co., a subsidiary of the Standard interests. The company continues to develop its properties in the Palo Seco, Fyzabad and La Brea districts. A refinery to treat its products has been constructed recently by the West India Oil Co. and is now in operation.

Fyzabad Dome Oilfields.—During the early part of the period under review a local operator commenced drilling on lands in the Fyzabad area and obtained a considerable production. Unfortunately, one of the wells came in out of control and was set on fire, resulting in the death of the owner, the driller and a number of others present at the time, a total of sixteen persons in all.

Barbados.

Commercial production has not yet been obtained in this colony.

British Guiana.

Whilst drilling for water in this colony some months ago it was reported that oil shows were met in one of the wells. The well was plugged immediately to avoid any possible flow which could not be controlled by the existing plant. Since then it is understood that there have been no active operations for the production of oil.

The Polish Oil Industry.*

By S. Pilat, Ph.D. (Member).

It can be observed that in recent years the oil-refining industry has been forced by the decreasing output of the Polish oilfields to improve the old-fashioned methods to the farthest possibility and to investigate some technical problems which were of a special importance for its working conditions. Some of these problems, as well as some of the solutions, having been adopted at the same time, are probably of a more than only local interest, and their examination may enable us to draw up the lines of the future development of this industry to a certain extent.

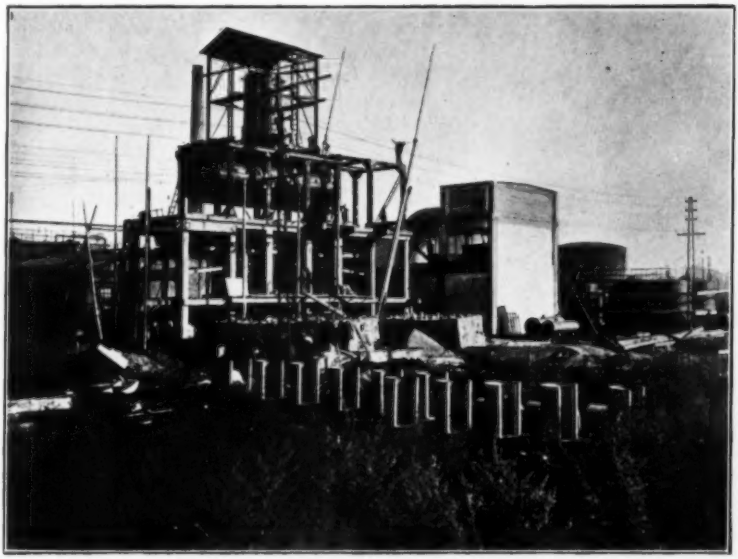
Among the problems which were to be solved carefully in order to retard the decrease of the crudel oil output in the district of Boryslaw-Tustanowice there was as a chief one, and one of an outstanding actuality, the decomposition of crude oil emulsions. Here it may be mentioned that there is a large and still increasing proportion of oil lifted in emulsion form, and the necessity of oil recovery from these emulsions can be considered as of vital importance to our industry.

In 1917 Prof. Moscicki, the actual President of the Polish Republic, worked out a method of breaking up emulsions by heating them in tightly-closed stills to a temperature of 130–140° C. At this temperature the emulsion breaks up easily and without any losses, and the recovered oil does not contain more than 0.3 per cent. of water. In the course of the same year this method was applied on a commercial scale, using first pressure-proof batch-stills, and then in continuously working apparatus, with most satisfactory results even where other processes could not be successfully adopted. This method of oil recovery does not exclude the simultaneous use of chemicals, such as naphthenic acids, phenols, etc., in order to effect the breaking up of the emulsion with an increased speed. It may be noted that the heat exchanger between the cold emulsion and the dehydrated hot oil which left the apparatus was so arranged that there could be obtained a high thermal efficiency with this process.[1]

Another problem of importance was that of gasoline recovery from natural gas. The natural gas from the main oil district of Boryslaw-Tustanowice contains on the average 40–50 g/cm. of gasoline, and the total of actually recovered gasoline is somewhat about 2,500 tons a month. Most of this gasoline is recovered

* Paper received January 13, 1930.

[1] Burek *Przem. Chem.*, 1923, 7, 281.



STEINSCHNEIDER DISTILLATION PLANT UNDER CONSTRUCTION AT JASLO, POLAND.

STEINSCHNEIDER DISTILLATION PLANT UNDER CONSTRUCTION AT JASLO, POLAND.

according to the proceeding of the Carblounion Co., Frankfurt-on-Main (Germany), in which activated charcoal produced by Bayer & Co. in Leverkusen (now J.G. Farbenindustrie) is used. This proceeding had been adopted in Poland for the first time in 1922, and was found so efficient that about 80 per cent. of the total gasoline is recovered by it.[2]

During the first period of introduction of this method into Poland an interesting, although not very well known experiment, had been made, which led to a certain degree to the better knowledge of the mechanism of such processes. An attempt has been made not to distil out the absorbed gasoline with the aid of steam, but by using a superheated inert gas which has been kept in continuous circulation by means of fans. But if the recovery of gasoline from charcoal is done with an inert gas it requires so high temperatures, that in dealing with a product of an end point of more than 180° C. an appreciable decomposition is already taking place, a decomposition which naturally affects the absorptive power of the charcoal.

Some practical improvements were to be expected from a recently suggested modification of the actual working scheme, which enabled higher average absorption rates of the charcoal to be obtained by changing the direction of the flow of gases[3]. On using activated charcoal for the purpose of analytical determination of the gasoline contents in a natural gas, we have the possibility of taking the specific gravity of it before and after the absorption, and the difference of these figures gives the amount of gasoline in the volume unit. Thus, we avoid the necessity of measuring the gas volume, which is of great importance for technical work. Care must be taken, however, not to reach the saturation point of the used charcoal.

Comparative experiments on commercial scale with liquid absorption agents, as for example tetraline, led to a further development of the charcoal plants. The efficiency of the charcoal process is best illustrated by the fact that for each kg. of activated charcoal about 1·4 kg. of gasoline can be daily produced. The steam consumption is hardly more than 2 kg. per 1 kg. of gasoline.

Important quantities of dry gas are used as fuel. This gas is distributed by means of pipe-line systems. The length of these lines, which is still increasing, amounts already to 250 km. for the main lines only.

Although no gas is used at the present time as a primary matter for chemical purposes, some industrial possibilities were found by experiments, which were conduced lately at the Polytechnic Institute at Lwow by Prof. Sucharda. It has been shown by

[2] Pol. Pat. 2,301.
[3] S. Pilat, E.P. 295,874, 1927.

this work that methane, or natural gas with about 95 per cent. of methane, can be easily chlorinated by passing it into a mixture with air and hydrochloric gas through heated tubes containing some cupric chloride as catalyst[4]. The reaction takes place quite easily and smoothly, and there is no danger of any explosions which seriously handicapped all the previous works on this subject. The chlorinated products are of a remarkable degree of purity, which permits a commercial utilisation of this method. Further improvements of the chlorination process, worked out by the same author, consist in conducting the operations in several stages, the products being separated from the gaseous reaction mixture after each stage. The simultaneous use of chlorine and hydrochloric gas for the identical process has been successfully applied.

The use of natural gas for the production of hydrogen has been studied by S. Mantel, who succeeded in producing by thermal decomposition at 1700° C. hydrogen with 0·7 per cent. of methane only.[5] The cracking of the propane and butane fraction of the natural gas led J. Winkler to olefinic and aromatic hydrocarbons, among which naphthalene and anthracene were found. The main product of the reaction at 730° C. were olefinic hydrocarbons mostly propylene, which can be converted into isopropylalcohol, thus increasing the alcohol production from gases of the normal cracking process.[6]

The possibility of conversion of higher olefines into corresponding secondary and tertiary alcohols with satisfactory yields has been demonstrated recently, contrary to the statements of Ormandy and Craven.[7]

The refining methods used by the Polish refineries were suitably adapted to the quality of the crude oil mainly produced in Poland. As mentioned before, this oil comes from the district of Boryslaw-Tustanowice, and the average yields of fully refined commercial products are as follows :—

	%
Naphtha	11
Kerosine	30
Gas oil	22
Lubricating oils	16
Paraffin wax	8
Coke	3
Operating loss	10
Total	100

[4] E. Sucharda, Pol. Pat. 10,462, 1929.
[5] S. Mantel, *Przem. Chem.*, 1928, **12**, 333.
[6] W. J. Piotrowski and J. Winkler, *Przem. Chem.*, 1930, **14**, 49.
[7] S. Pilat and J. Winkler, *Przem. Chem.*, 1929, **13**, 186, 209; E. Holzman and S. Pilat, *Przem. Chem.*, 1929, **13**, 455; Ormandy and Craven, *Chem. and Ind.*, 1929, **48**, 293.

As paraffin wax is one of the most valuable products, and considering the necessity of its extraction with regard to the quality of the lubricating oils, most of the Polish refineries are equipped with efficient wax plants. In order to obtain high yields of paraffin wax the crude is distilled as a rule to coke. The chief aim of the distilling operations is to avoid any decompositions by cracking, which would affect the yield of paraffin wax and the quality of the lubricating oils. Continuously working shell-still plants are much in use. In these the crude oil is stripped of 50-60 per cent. of lighter parts, *i.e.*, naphtha, kerosine, and some light paraffin oil. The residue is distilled in separate vacuum distilling stills, working at a pressure of about 60 mm. mercury, an operation which is carried on by using a fair amount of steam. The result of this is that the main quantity of wax-distillate is taken out while the asphalteous residue, which is distilled to coke in small batch stills by using large quantities of steam, prevents the decomposition of the wax.

The main products of these rather complicated operations are naphtha distillate, kerosine, wax distillate and coke. The yield of wax-distillate amounts to 45–50 per cent. of the crude oil.

A progress worth noticing was accomplished during recent years through an extensive application of vacuum plants for wax distillates. The very important and rather striking result was the increase of the yield of paraffin wax from 6 to 8 per cent. and even more of the crude oil worked up. A recently constructed distilling plant designed by Mr. L. Steinschneider, and executed by the Brünn-Kœnigsfelder Co. in Brünn, Czecho-Slovakia, is working on these lines. It permits the continuous distillation of wax by using at the same time two stages of vacuum, one of 25 and one of 5 mm. pressure. This plant, which seems to be one of the most efficient of its kind, was described in *J. Inst. Petr. Tech.*, 1925, **11**, 514-523. During trial runs on commercial scale 1 mm. mercury pressure could have been maintained no steam being used, and the resulting distillate was absolutely dry and clear. This high-vacuum distillate when treated with sulphuric acid has lost much less of its viscosity than corresponding products from the ordinary low-vacuum distilling plant. For example, the viscosity of low-vacuum plant distillate of about 20 Engler at 50° C. drops after treatment to 14 at 50° C. With the high-vacuum distillate of a viscosity of only 16 at 50° C. results the same oil of 14 viscosity after treatment.

The technical advantage of the use of a pipe-still for heating the oil or residue to the required temperature and the shell-still as evaporator is much too obvious to be discussed.

The direct separation of wax from the crude oil itself, without

distillation, was the main object of numerous experiments and investigations, but until now no commercial success could be obtained. However, a promising step in this direction may be noted. It has been observed by E. Katz that paraffin wax may be separated from oil, using its faculty of forming mixed crystals with some solid and easily crystallising hydrocarbons of the aromatic series[8], and some very high melting wax was produced in laboratory experiments by this method. The filter cake produced by this method in a semi-commercial experimental plant cannot be worked up by the usual sweating process, the wax being of a rather amorphous form. Anyhow the cake is so far free from oil that after a recrystallisation from a suitable solvent (kerosine) and steaming which removes the naphthalene, high-melting wax can be easily produced. This sort of wax of a m.p. above 70° C. is a product similar to ozokerite.

Among the improvements in treating lubricating oils, it may be pointed out that most of the refineries are using now different concentrations of sulphuric acids, and that mostly the 85–90 per cent. is used instead of the 95–96 per cent. (66° Bé) which had been used exclusively for years before. The satisfactory effect consists chiefly in a diminution of the treating loss.

The treatment of lubricants with considerable quantities of chemicals is closely connected with the important question of utilising the by-products resulting from these operations, a question most interesting from the chemical view as well as from the commercial one.

Although most of the investigations related to those problems are still carried out on laboratory scale, some of them have found already a commercial application.[9] The starting point for this interesting work has been the good result of the method of breaking up emulsions by heating them under pressure in the same way as it had been done with crude-oil emulsions. Those experiments were as far successful as suitable concentrations were chosen to break up the emulsions readily, and from the alkaline waters sodium salts of sulpho-acids could be recovered by saturation with salt.

A closer examination of these sulpho-acids gave their general formula as $C_{22}H_{18}SO_3H$. They are dark, viscous, half-solid substances, heavier than water, and very hygroscopic. They are soluble in water, and excellent emulsifiers, and their aqueous solutions are much used in the Polish textile industry. It may be of some interest to note that these bodies are almost similar, if

[8] E. Katz, Pol. Pat. 11,267, 1929.

[9] Pol. Pat. 2,958, 1922. S. Pilat and E. Davidson, *Przem. Chem.*, 1927, **11**, 141; *Petroleum*, 1928, **24**, 1559.

not identical, with those which were produced for similar purposes by the Russian oil industry, and brought into market under the name of "Contact." Although the Russian product was obtained by the action of highly concentrated and fuming sulphuric acid, while ours resulted from the treatment with 85–90 per cent. acid only, it can be said that the chemical effect in both cases was nearly the same.

The molecular weight of those acids is about 410, but there were found subsequently two groups of sulphonic acid not mentioned before with lesser molecular weights. Both of them proved to be oxy-sulphonic compounds, and that one of them a mono-oxy-sulphonic acid of a molecular weight of about 370, easily soluble in water, and copiously foaming, and the other one a dioxy-sulphonic acid of a molecular weight of about 270. The latter substance is very easily soluble in water, and cannot be precipitated from its aqueous solutions even by fairly concentrated acids. The aqueous solution shows a beautiful green fluorescence.

The investigations mentioned above make it most probable that the future development of the Polish petroleum industry will follow the chemical side of the refining processes more closely than in the past.

THE INSTITUTION OF PETROLEUM TECHNOLOGISTS.

ANNUAL REPORT OF THE COUNCIL, 1929-30.

THE Accounts for the Year ended December 31st, 1929, together with the Annual Report of the Council for the Session, 1929-30, are hereby submitted.

The table given below shows the total membership as at December 31st and the changes during the year. It will be seen that the Membership of the Institution maintains its steady increase.

	TOTAL 31.12.28.	CHANGES DURING 1929.							TOTAL 31.12.29.
		New	Transferred to.	Re-signed.	Deceased.	Removed.	Transferred from.	Net gain.	
Honorary Members	14	3	—	1	1	—	—	1	15
Members	511	33	12	7	8	4	—	26	537
Associate Members	392	39	16	6	—	5	12	32	424
Students	117	22	—	2	1	7	10	2	119
Associates	81	11	—	2	—	3	6	—	81
TOTALS.....	1115	108	28	18	10	19	28	61	1176

The Accounts for the year ended December 31st, 1929, are presented and Council are glad to state that the Balance Sheet continues to show a satisfactory financial position.

The Scholarships awarded by the Institution have been provided for in the Accounts and it has been decided to continue the Awards for the year 1930.

The revenue from Members' Subscriptions shows a satisfactory advance.

The publication of the second edition of "Standard Methods of Testing Petroleum and Its Products" was a noteworthy feature of the session. The large demand for this book indicates its value to the industry, and the Council are indebted to the Members of the Standardization Committee for the valuable services rendered in its preparation.

The Institution had representatives on The American Society for Testing Materials; the Advisory Board of the Department of Oil Engineering and Refining of the University of Birmingham; the British Association for the Advancement of Science; the British Engineering Standards Association; the Fuel Economy Committee; the Imperial Mineral Resources Bureau; and the Mechanical Warfare Board of the War Office.

Official Delegates were also appointed and attended the 2nd International Drilling Congress (Paris) and the 9th Congrés de Chimie Industrielle (Barcelona). A Complimentary Address was sent to the World Engineering Congress, held in Tokyo in October, 1929.

The Institution held eight General Meetings during the year 1929 and the following papers were read and discussed :—

One Hundred and Sixteenth General Meeting, January 8: "Geophysical Surveying," by W. H. Fordham.

One Hundred and Seventeenth General Meeting, February 12: "The Chemical Investigation of Trinidad Well Waters and Its Geological and Chemical Significance," by J. S. Parker and C. A. P. Southwell.

One Hundred and Eighteenth General Meeting, March 12: "Drilling for Oil with the Diamond Drill," by G. Heseldin.

One Hundred and Nineteenth General Meeting, April 9: "The Oilfields of Burma," by L. Dudley Stamp.

One Hundred and Twentieth General Meeting, May 14: "Flame Characteristics of 'Pinking' and 'Non-Pinking' Fuels," Part II., by G. B. Maxwell and R. V. Wheeler.

"The Influence of Cylinder Design on Pinking," by G. B. Maxwell and R. V. Wheeler.

One Hundred and Twenty-first General Meeting, October 8: "The Estimation of Gum in Petrol and Its Significance," by S. J. M. Auld.

"The Deterioration of Cracked Spirits by Gumming," by E. W. J. Mardles and H. Moss.

"Gum Formation in Cracked Gasolines," by C. R. Wagner and Julius Hyman.

"The Determination of Existent Gum in Cracked Distillates," by W. S. Norris and F. B. Thole.

One Hundred and Twenty-second General Meeting, November 12: "Oil Legislation in Central and South America," by Sir A. T. Wilson.

One Hundred and Twenty-third General Meeting, December 10: "The Engineer in Relation to the Petroleum Industry," by John Gillespie.

Volume 15 of the Journal was published in six parts during 1929 and contained the papers read at the meetings, together with their discussions, and also articles dealing with the various phases of the Petroleum Industry. The Abstract section of the Journal contained nearly one thousand Abstracts of the more important literature and patent specifications of interest to petroleum technologists. In order that the Abstracts and the papers contributed to the Journal may reach its readers with the minimum of delay, it has been decided that from 1930 the Journal shall be published in nine parts per annual volume.

The London and Birmingham Branches of the Students' Section report the holding of regular meetings and the reading of papers,

The Redwood Medal for the Sessions 1927-28 and 1928-29 was awarded to Mr. W. H. Fordham (Member) and the Student's Medal and Prize for the Session 1928-29 was gained by Mr. H. M. Stanley.

The Scholarships were awarded to Mr. L. C. Stevens and to Mr. J. J. G. Clark at the Birmingham University and at the Royal School of Mines respectively.

The Institution's Research Fellowship, founded during the current year, has been awarded to Dr. H. R. Lang, who is carrying out research work at the Imperial College on Specific Heats.

Dr. A. E. Dunstan, D.Sc., F.I.C., F.C.S., has been unanimously re-elected President for the ensuing Session, and Sir Richard Barnett, Sir John Cargill, Mr. Alexander Duckham, Mr. Arthur W. Eastlake, Mr. James Kewley and Mr. Robert Redwood have been unanimously re-elected as Vice-Presidents, and Mr. A. E. Chambers has been unanimously elected as Vice-President for the ensuing year.

The Council have most carefully considered the making of an application to His Majesty's Privy Council for the granting of a Royal Charter to the Institution, and as a referendum of the Members showed virtual unanimity the matter is being proceeded with.

The Report of the Library Committee shows that increasing use is being made of the Library and that not only Members of the Institution, but Government Departments and industrial organisations, both in this country and abroad, find it a ready source of information. The Library Catalogue is divided into three sections, Geographical, Subjects and Authors, the first two being sub-divided into appropriate groups.

The Institution's Branches in Rumania, Persia and Trinidad continue to make satisfactory progress, and a new one has recently been established in South Wales.

The Council desire to call attention to the recent foundation of a Benevolent Fund, inaugurated by the President, regarding which a letter was circulated to all Members. A Banker's Order, for the convenience of those wishing to contribute, was included with the December, 1929, issue of the Journal, and it is hoped that members will subscribe generously and regularly to the Fund.

The Eleventh Annual Dinner was held at the Connaught Rooms on October 11, 1929, and was attended by over 200 members and guests. On December 19, 1929, a Conversazione was held at the House of the Royal Society of Arts, and, by the courtesy of Mr. James Kewley, a most interesting film of operations in the Petroleum Industry was shown to a gathering of about 130 Members and their friends.

It is with regret that the Council have to record the deaths of the following members: T. O. Bosworth, F. D. Brown, Sir A. H. Burgoyne, W. H. Dalton, H. Y. V. Jackson, A. G. Leighton, M. Lodge, C. H. McCarthy Jones, H. Pearson and F. Russell.

The Royal Society of Arts has continued to allow their House to be used for the meetings of the Institution and the Chemical Industry Club has permitted the Members of the Dinner Club to dine at its premises. To both these bodies the Council desire to express their thanks and appreciation of their courtesy.

The following have again rendered signal service to the Institution and to them the Council present their sincere thanks: Sir William Plender, Bart., G.B.E., Honorary Treasurer; Messrs. Ashurst, Morris, Crisp & Co., Honorary Solicitors; Messrs. Price, Waterhouse & Co., Auditors; Sir Frederick W. Black, K.C.B., Honorary Editor; Mr. George Sell, Assistant Editor and Secretary to the Standardization Committee; Commander R. E. Stokes-Rees, R.N., Secretary; Miss C. Despard, Librarian; and the Office Staff at Aldine House.

The Auditors retire, and being eligible, offer themselves for re-election.

By Order of the Council,

ARTHUR W. EASTLAKE,

Honorary Secretary.

Dr. REVENUE ACCOUNT FOR THE

	£	s.	d.
To Salaries	1086	7	0
„ Rent, Rates, etc.	440	10	4
„ Printing and Stationery	173	4	6
„ Postage	210	5	8
„ Expenses of Meetings, Including Hire of Hall	209	9	0
„ Journals and Publications (Net Expense)	379	13	5
„ Grants to Branches	128	0	0
„ Sundry Expenses	298	11	9
„ Auditors' Fee	10	10	0
„ Students' Prize and Scholarship Fund	80	0	0
„ Research Fund	100	0	0
„ Depreciation of Office and Library Furniture and Library Books	50	8	11
„ Deficit on Students' Section Fund Transferred	1	11	7
„ Balance—being Surplus for the Year Carried to Balance Sheet	4	3	7
	£3172	15	9

STUDENTS' PRIZE AND

1929.		£	s.	d.	£	s.	d.
Dec. 31.	To Scholarships Granted—						
	Imperial College of Science and Technology	40	0	0			
	Birmingham University	40	0	0			
					80	0	0
	„ Balance Carried Forward				10	0	0
					£90	0	0

RESEARCH

1929.		£	s.	d.
Dec. 31.	To Advertising Fellowship	10	8	6
	„ Fellowship and College Fees	124	10	0
	„ Balance Carried Forward	1054	3	8
		£1189	2	2

STUDENTS'

1929.		£	s.	d.
Dec. 31.	To Expenses for the Year	17	0	10
		£17	0	10

YEAR ENDED 31ST DECEMBER, 1929. Cr.

	£ s. d.	£ s. d.
By SUBSCRIPTIONS—		
Members	2987 5 0	
Special	20 0 0	
		3007 5 0
„ INTEREST AND DIVIDENDS LESS TAX		165 10 9
		£3172 15 9

SCHOLARSHIP FUND.

1929.		£ s. d
Jan. 1.	By BALANCE BROUGHT FORWARD	10 0 0
	„ REVENUE ACCOUNT	80 0 0
		£90 0 0

FUND.

1929.		£ s. d.
Jan. 1.	By BALANCE BROUGHT FORWARD	996 8 11
Dec. 31.	„ INTEREST AND DIVIDENDS	30 13 3
	„ REFUND OF AMOUNT UNEXPENDED FROM PREVIOUS GRANT	62 0 0
	„ REVENUE ACCOUNT	100 0 0
		£1189 2 2

SECTION FUND.

1929.		£ s. d.
Jan. 1.	By BALANCE BROUGHT FORWARD	15 9 3
Dec. 31.	„ BALANCE DEFICIT ON FUND TRANSFERRED TO REVENUE ACCOUNT	1 11 7
		£17 0 10

BALANCE SHEET AS

	£ s. d.	£ s. d.	£ s. d.
CAPITAL OF THE INSTITUTION, under Bye-Law Section 6, paras. 14 and 15 :—			
Life Membership Fund—			
As at 31st December, 1928	451 5 0		
Additions during year	47 5 0		
		498 10 0	
Entrance Fees—			
As at 31st December, 1928	1679 19 0		
Additions during year	143 16 0		
		1823 15 0	
Profit on Sale of Investment—			
As at 31st December, 1928		2 10 0	
Donations—			
As at 31st December, 1928		326 5 0	
			2651 0 0
STUDENTS' PRIZE AND SCHOLARSHIP FUND			10 0 0
RESEARCH FUND			1054 3 8
MEMBERS' SUBSCRIPTIONS PAID IN ADVANCE			110 5 0
JOURNAL SUBSCRIPTIONS PAID IN ADVANCE			341 17 7
SUNDRY CREDITORS			379 7 7
REVENUE ACCOUNT :—			
Balance as at 31st December, 1928 ..		1651 8 4	
Less : Unpaid subscriptions for 1928 written off		56 14 0	
		1594 14 4	
Add : Subscriptions previously written off and now collected..		16 16 0	
		1611 10 4	
Add : Surplus for year as per separate Account		4 3 7	
			1615 13 11
			£6162 7 9

Members of Council { ARTHUR W. EASTLAKE.
J. KEWLEY.

We have examined the above Balance Sheet with the books of the Institution are of opinion that such Balance Sheet is properly drawn up so as to exhibit a of our information and the explanations given to us and as shown by the books

3, FREDERICK'S PLACE,
OLD JEWRY, LONDON, E.C. 2.
11th *February*, 1930.

AT 31ST DECEMBER, 1929.

	£ s. d.	£ s. d.
INVESTMENTS AT COST :—		
On Account of Capital—		
£1250 0 0 5% War Loan 1929/47	1196 18 6	
£650 0 0 3½% Conversion Loan	505 7 0	
£664 6 6 London County 3% Consolidated Stock	481 10 6	
£150 0 0 Birmingham Corporation 6% Stock 1936/46	146 16 0	
£100 0 0 Wigan Corporation 5% Redeemable Stock	99 14 11	
£160 0 0 New South Wales 5% Stock 1945/65	158 7 0	
		2588 13 11
(Market Value at 31st December, 1929, £2535 19s. 9d.)		
On Account of Revenue—		
£1100 0 0 5% War Loan 1929/47		1118 3 6
(Market Value at 31st December, 1929, £1102 15s. 0d.)		
On Account of Research Fund—		
£500 0 0 5% War Loan 1929/47		510 8 3
(Market Value at 31st December, 1929, £501 5s. 0d.)		
OFFICE AND LIBRARY FURNITURE (excluding Presentations) :—		
As at 31st December, 1928	450 3 3	
Additions during year	0 19 6	
	451 2 9	
Less : Depreciation	22 10 2	
		428 12 7
LIBRARY BOOKS (excluding Presentations) :—		
As at 31st December, 1928	186 4 11	
Additions during year	15 19 6	
	202 4 5	
Less : Depreciation	27 18 9	
		174 5 8
MEMBERS' SUBSCRIPTIONS FOR 1929 OUTSTANDING CONSIDERED COLLECTIBLE		114 3 6
SUNDRY DEBTORS AND PAYMENTS IN ADVANCE ..		148 1 6
CASH ON DEPOSIT WITH BANKS :—		
Research Fund	353 1 11	
General Account	400 0 0	
		753 1 11
CASH AT BANK AND IN HAND		326 16 11
		£6162 7 9

and have obtained all the information and explanations we have required. We true and correct view of the state of the Institution's affairs according to the best of the Institution.

PRICE, WATERHOUSE & CO.

THE INSTITUTION OF PETROLEUM TECHNOLOGISTS.

SEVENTEENTH ANNUAL GENERAL MEETING.

THE SEVENTEENTH ANNUAL GENERAL MEETING of the members of the Institution was held at the House of the Royal Society of Arts, John Street, Adelphi, London, on Thursday, March 20th, 1930. The President, Dr. A. E. Dunstan, was in the Chair.

The Secretary (Commander R. E. Stokes-Rees, R.N.) read the notice convening the meeting and the Minutes of the last meeting.

The Minutes having been signed, the Secretary announced the result of the ballot for the Council. He stated that there were five nominations for the Council, three of whom were retiring members, and two new names. The three retiring members who had been re-elected were Dr. F. B. Thole, Mr. T. R. H. Garrett and Mr. A. Frank Dabell, and Dr. F. H. Garner was elected.

It was agreed that the list of Members, Associate-Members, Students and Associates elected and transferred during 1929 be laid on the table.

The Secretary read the list of new members nominated and elected since the last General Meeting.

At the Council Meeting, held on February 11th, 1930, the following gentlemen were nominated :—

Transfer to Member.—Douglas Leonard Alexander, Eric James Dunstan.

Associate Members.—William Duff, Edward Vernon Wharton.

Transfer to Associate Member.—Hendrik Willem Slotboom.

Students.—Jack William Clack, Samuel Elliman, Kenneth Byrne Lanyon.

At the Council Meeting, held on March 20th, 1930, the following gentlemen were nominated :—

Members.—David Allan, Baron Henry M. de Cizancourt, Robert Crichton, Paul D. Foote, Leon Iacovitz, Richard Henry Thorburn Jobson, Alexander Reid, Alexander Walker.

Transfer to Member.—Harvey Satchell Garlick, Charles Ignatius Kelly.

Associate Members.—John William Amner, Edgar Beardsworth Fox Auld, Vincenzo Damiani-Lugaro, Robert Hugh Dolton, Edward Leslie Gent, John Harry Maltby, Horace Edward Newton, Andre A. Perebinossoff, Hugh Charles Tett.

Transfer to Associate Member.—John Wardlaw Reid.

Students.—Walter William Scott, Thomas Speight, Joseph Edwin Walker.

Associate.—Hugh Peregrine Henzell, Frederick Caulfield Kearney, John Macdonald, Clarence Achilles Palmer-Chizzola, William Sigsworth, Henry David Thom.

At the Council Meeting, held on March 20th, 1930, the following gentlemen were elected :—

Members.—Donald Charles Broome, William John Burchett, Douglas Harold Jackson, Arthur Whittaker Richardson.

Transfer to Member.—Henry Frederick Moon.

Associate Members.—Henry Norman Bassett, John Howard-Glossop Goodfellow.

Transfer to Associate Member.—David Glynn-Jones.

Students.—Reginald Charles Rogers, Herbert William Harry Wilkinson.

The Secretary then read the Annual Reports of the London and Birmingham Branches of the Students' Section.

London Branch.—At December 31st, 1929, the Students of the Institution numbered 119. During the year under review there were, in the London area, 15 new members, to whom a hearty welcome is extended; 2 members were transferred, 5 removed, 1 resigned, and we regret to say 1 killed in a motor accident.

At the Sixth Annual General Meeting, held in February, 1929, the following were elected to serve on the Committee: Messrs. J. E. Duckham, W. B. Rowntree, G. Paice, C. E. Hobley, J. J. G. Clark and P. A. Spens. Mr. Duckham was subsequently elected Chairman; Mr. Spens, Honorary Secretary; and Mr. Rowntree, Honorary Excursion Secretary.

During 1929, seven meetings were held :—

January, 1929: "The Mechanical Testing of Motor Spirit," by W. A. H. Oswald, B.A., F.C.S. (Associate Member).
February, 1929: "White Oils," by G. Coles.
April, 1929: "The Anglo-Persian Oil Film," shown by W. B. Rowntree (Student) and A. S. Gray, B.Sc.
May, 1929: "Low Temperature Carbonisation," by G. H. Willock (Member).
October, 1929: "Some Characteristics of Mineral Lubricating Oils," by P. Driver (Associate Member).
November, 1929: "Crank Case Dilution," by H. E. Tester (Student), read by W. B. Rowntree.
December, 1929: "Pinking," by N. Wood, B.Sc., F.C.S. This unfortunately had to be postponed by lack of attendance.

Two excursions were held during 1929 :—

February, 1929 : To Messrs. Baird and Tatlock (London), Ltd., factory.
December, 1929 : To the Commonwealth and Dominion's Motor Ship the M.V. "Port Alma."

The attendance at these was fairly good.

At the meetings during the past year, the attendance has been appallingly bad. Two letters demanding better attendance have been sent out during the past year. This is not in accordance with the prestige of the Students' Section. Too many Students seem to think that their work ends with paying £1 1s. a year. We hope that it will be better during 1930.

Six student's papers were entered for the Annual Medal and Prize offered by the Institution. Of these, three were from the London area. The Council awarded the Medal and Prize to Mr. H. M. Stanley, of Birmingham, for his paper on "Recent Advances in Our Knowledge of Natural Gas and 'Cracking' Gas and Their Industrial Utilisation." There has been a lamentable lack of Student's papers during the past year, and we sincerely hope that there will be more during 1930.

The Committee takes this opportunity of thanking the senior Members of the Institution who have attended meetings and those who have assisted in furthering the activities of the Students' Section throughout the year.

(Signed) P. A. Spens, Hon. Secretary.
(Countersigned) A. E. Dunstan, President.

Birmingham Branch.—The Committee has pleasure in presenting the Seventh Annual Report, for the year ended December 31st, 1929. During the year seven meetings have been held, at which the attendance has been very good.

The following papers were read :—

January 22nd, 1929 : "Oilfield Emulsions," by Donald A. Howes, M.Sc. (Student).

February 12th, 1929 : "Recent Advances in Our Knowledge of Natural Gas," by H. M. Stanley, M.Sc. (Student).

March 5th, 1929 : Recent Improvements in Oilfield Equipment," by R. B. Rogers (National Supply Corporation).

March 12th, 1929 : Annual General Meeting. Election of Officers for 1929-30 Session.

May 7th, 1929 : "Methods of Correlation of Underground Sands and Waters," by W. R. Wiggins (Student).

October 29th, 1929 : "Internal Combustion Locomotives," by P. N. D. Porter (Student).

December 11th, 1929 : "Mechanical Design in Relation to Compression Ratio," by A. C. Chamberlain (Student).

On November 22nd, 1929, a visit was paid to the Openshaw Works of the English Steel Corporation, when their latest type of Rotary rig was seen in action.

Three suppers were also held, at which a large proportion of members attended.

(Signed) Launcelot P. Timmins, Hon. Secretary.
(Countersigned) A. E. Dunstan, President.

The President said the next business was to submit the following resolution :—

"That this meeting of the members of the Institution of Petroleum Technologists hereby approves of the presentation to His Majesty the King in Council of a Petition for the grant of a Royal Charter to the Institution."

He said the matter had been considered exceedingly carefully. It was by no means a new feature of their deliberations, because it was always felt in the past that the Institution could very well wait for a membership of 1,000 before putting forward an appeal for a Royal Charter. The membership was now a long way beyond 1,000—in point of fact, not far short of 1,300. The Council, with the assistance of Honorary Solicitors and a special committee, had gone into the matter in great detail, and he therefore moved the resolution which he had read.

Mr. Arthur W. Eastlake seconded the resolution, which was carried unanimously.

The President moved the adoption of the annual report of Council for 1929-30. He said he would take the report itself and leave Mr. Adams to deal with the financial side. Allusion might be made to one or two points. Firstly, the Institution's scholarships had been continued. No one need emphasise the vast importance of that side of their activities. The publication of the second edition of "Standard Methods of Testing Petroleum and its Products" had been a feature of the past year, and the demand for that monograph had certainly exceeded all the anticipations which the Council had when they put it forward. The Council would wish to thank those gentlemen who had put in so much work in connection with the compilation of the standard tests—Professor J. S. S. Brame, Sir Frederick W. Black and Mr. G. Sell had done yeoman service in that direction.

It would be seen by the report the wide representation the Institution had on various societies. Almost day by day the influence and activities of the Institution spread.

Coming to the Journal, the outstanding feature was the decision to publish nine numbers annually in place of six hitherto. That inevitably was going to throw a great deal more work on those concerned in its preparation. Last year the abstract section contained nearly 1,000 abstracts of the more important literature and patent specifications of interest to petroleum technologists. Everyone agreed that no effort must be spared to make the Journal even better than it was to-day, and he was sure all would agree with that view. The meeting had heard the account of the students' branches. He did not know whether anything

could be done to supplement what Mr. Adams told them last year as to the disappointing attendance. Possibly if more senior members attended it would be an encouragement to the students to come forward.

The Institution's Research Fellowship had been awarded to Dr. H. R. Lang, who was carrying out research work, at the Imperial College, on Specific Heats. The Institution's branches were most effective. The Persian, Rumanian and Trinidad branches met regularly and discussed matters and sent in material for the Journal. It had been an exceedingly successful experiment; and they had to record the formation of a new branch in South Wales.

The dinner was held with great success in October, and their best thanks were due to Mr. Ashley Carter for the work he put in in that direction. The report wound up, as was common, with an omnibus vote of thanks which he was perfectly certain would be accepted in the spirit in which it was put forward.

Sir Richard Barnett seconded the motion for the adoption of the Report.

Dr. A. Wade observed that he noted with regret what was in his opinion one very serious omission from the report. He thought the members should be taken into the confidence of the Council with regard to the proposal about which they heard only from the Press, namely, that the Institution should occupy premises somewhere jointly with other societies. They had heard nothing officially, but every now and then they read something in the newspapers and technical journals, though nothing had come from the Council. He thought the members themselves would like to consider the proposal since it might seriously affect the individuality of the Institution, and there were many problems in connection with the scheme which the members might assist the Council in considering.

The President, in reply, pointed out that the Council had had nothing whatever to report to the members. It was true that negotiations had been carried out. A number of societies had come together and more or less considered the general scheme before them, but before anything was decided it would come before the members. There was no hole-and-corner mystery about it, and as a matter of fact there was to be a public appeal made in the near future. The Council without all the facts before them were not in a position to discuss the matter.

The Report was adopted.

The Secretary read the auditors' certificate.

The President called on Mr. Adams to give as usual a summary of the balance sheet, after which he (the President) would propose it formally from the Chair.

Mr. A. C. Adams said that having been reappointed chairman of the Finance Committee after two years' absence from that position while he was President, it was his privilege once more to talk to them on the balance sheet and accounts. They were presented in what he thought all would agree was in very simple fashion. They had always been presented in that way, and there was really little necessity for explanation as members were familiar with the form in which they were presented. If they turned to the balance sheet, which was the most important part of the accounts, it would be seen that the capital of the Institution had increased by about £190. That was due to life membership subscriptions and entrance fees which had to be invested and regarded as capital. On the other side, it would be seen that investments on account of capital amounted to £2,588 odd, and was within about £70 of the amount against the capital item. It would always be found that as these entrance fees and other matters accumulated it was necessary to wait a little while in order to have a sum invested, and their practice was to invest as soon as they had accumulated £100. At the present time, however, the amount invested in the capital account was more than the capital item on the other side of the balance sheet, because they had additional entrance fees, and so the investments rather overloaded the other item in a contrary direction. The research fund stood at £1,054 3s. 8d. Some portion of the Fellowship Grant had been paid to the holder of the fellowship, but not all for a whole year, and Council had allocated to the Research Fund £100 out of its balance for the year 1929. The members' subscriptions were quite ordinary items, and the sundry creditors represented amounts which had not been actually paid at the end of the year.

Turning again to the other side they had invested on account of revenue £1,018 3s. 6d., and there was also invested, quite properly, he thought it would be agreed, a large amount of the Research Fund, so that it was there until it was wanted for the purpose of paying out for fellowship purposes. With regard to the Office and Library Fund, the library books had been depreciated—liberally depreciated because it was felt to be necessary to do so. The members' subscriptions for 1929 which were considered collectable were outstanding subscriptions which they hoped would come in; they were a little belated in some cases. It was their practice to write off unpaid subscriptions from time to time where it was thought they were unlikely to come in. On the other hand,

it was rather gratifying to find that they did come in sometimes and so they were brought in again. There was cash on deposit at the banks on account of Research Fund £353 1s. 11d. and on General Account £400. There was also cash at the bank and in hand £326 16s. 11d., and it would be agreed that that indicated quite a healthy position in regard to the finances of the Institution. Detailed particulars would be found of the Students' Prize Fund and Students' Section Fund which appeared below the Revenue Account. There was very little in the Revenue Account that called for particular attention except with regard to the item of Journals and Publications. Here a small departure had been made in that the net expense was given in connection with the Journals and all the other publications which the Institution issued including the Book of Standard Tests. The net amount which the whole of the publications cost the Institution in 1929 was £379 13s. 5d. That method was followed for a number of reasons which he need not particularise, but also to avoid possible confusion and the necessity for referring to each publication in a separate way which, after all, was not necessary. Grants to branches, £128, was an item which it might be naturally expected would become an increasing one as further branches were opened. They had put £100 to Research Fund and the resulting balance was £4 3s. 7d. There was no object in carrying forward £104 3s. 7d., so the Council thought it prudent to take care of the Research Fund and to augment it in that way. With regard to subscriptions, the Institution were not in the enjoyment of the very generous subscriptions they had had in previous years from the oil companies. It would be agreed that the position was satisfactory that they were able to make such a good showing without the benefit of those quite large amounts they had had in previous years. (Applause.)

The President said that in proposing the adoption of the balance sheet he was rather inclined to make it somewhat in the form of a vote of thanks to Mr. A. C. Adams. The Institution was particularly fortunate in having a Chancellor of the Exchequer whose careful watch over finance, considered judgment and wide experience in these matters was invaluable. He proposed the adoption of the balance sheet coupled with a vote of thanks to Mr. Adams. (Applause.)

The motion was carried unanimously.

The auditors, Messrs. Price, Waterhouse & Co., were re-elected on the proposition of **Mr. C. W. Wood**, seconded by **Mr. Fred Esling**.

The President then delivered his Presidential Address.

The Chemist in the Petroleum Industry.

By A. E. Dunstan, D.Sc., F.I.C., F.C.S.

It has been the custom at the annual meetings of this Institution for the President to be given a free hand in the selection and shaping of his discourse.

A variety of topics have been discussed during the past sixteen years, varying from the standardisation of tests to the equipment of a modern ocean installation.

I, in my turn, am going to speak as a chemist, and so far as may be, justify the presence of the chemist in our great industry and in our ever-growing Institution.

I do not propose to apologise for him, because I firmly believe that he is indispensable, and that the progress of our civilisation depends upon him: but I would emphasise that when he has become fledged, both in the practice of his profession and in the fellowship of the Institution, he becomes something more than a chemist—he becomes elevated to the rank of petroleum technologist. To borrow that famous motto, he as well as his fellow engineer and his fellow geologist, becomes *'e pluribus unum*—a member of a great profession, and his scientific entity becomes less important.

There are, of course, many types of chemists in the industry engaged in widely different fields of work, but they can be classified into three main groups depending on their academic qualifications when they start out in the industry, and their general make-up. These three groups are recruited as follows:—

(A) Young men straight from secondary school, who have passed, for example, the Intermediate Bachelor of Science examination.

(B) 1. Graduates of the Honours B.Sc. standard, and

2. Post-graduates M.Sc. or Ph.D.

The A class chemist should be appointed to a research or a routine laboratory in which he spends some years firstly on testing and analysis, and later as assistant to senior members of the staff, whilst working for his B.Sc. When this degree is obtained it is usual for him to proceed to a refinery at home or abroad, to gain further experience in works analysis, or on the operating side.

The A class men usually stay in the refinery, where they may rise to executive positions; there are, of course, exceptions, for when a man appears more than usually brilliant in his probationary service, he is often given special facilities to work for higher qualifications, and passes on to a higher grade.

The B class chemist is usually appointed direct to a research laboratory for analysis and general work, and after a preliminary period he is posted to the refinery as a development technician, or as a member of the refinery staff. This class of man, on first entering the research laboratory, is appointed to a definite line of investigation, depending upon the nature of his university record, and ability. He may then pass out to the refinery on specific development problems, or he may, in rarer cases, be retained for research.

I would wish at this stage to refer to the work of the analyst. It has often been assumed that his is the donkey-work of the chemical staff and that he is a " maid-of-all-work " in the laboratory. I would strongly controvert this assumption. In the first place the insistence upon accuracy that analytical chemistry demands is of prime importance to every type of chemist and indeed indispensable. Every process in the long run depends upon unerring analysis. Analysis is the backbone of the chemical service.

There is a further stage in the recruitment of the chemical service that I may refer to as class C. The recruit in this class cannot be absorbed as such, he is partly born and partly made. His is a case of nature and nurture. He arrives in category B., i.e., a thoroughly well-nurtured candidate, for whom his university has done her best. It is the function of his industrial training to afford him still further nurture, but his ultimate success will depend very greatly on his nature. And it is only after his probationary work under the watchful eye of his chief that he can be placed adequately. He must, if he is to qualify for this grade, be possessed of " plant sense." He must gain some measure of familiarity with large scale units. He may be regarded as an embryo chemical engineer.

Mention should be made here of the excellent training now to be had in the British universities. Not so long ago it would have been entirely impossible to take a British graduate in chemistry who had the faintest idea of plant. Even were he an honours man, and possessed of some research training, he left his college not only fully persuaded that his knowledge of chemistry was profound, but also very inclined to look down upon and to patronise men in the industry who were, in point of fact more than often his superiors in knowledge and in wisdom. This is largely changed. For one thing his professors are more likely than not in these days to have many commercial interests. The War took many charming academic gentlemen and pitchforked them into the hurly-burly of industrialism, and taught them that works chemistry differs from college chemistry mainly in that it is controlled by a time factor—a fourth dimension.

Professors of chemistry to-day are doing immense services to industry, and the newly-equipped departments of chemical engineering are giving us raw material of very definite value. At the risk of being accused of making invidious distinctions, I would wish to mention the departments at University College (The Ramsay Memorial Laboratories), at the Imperial College and at the Universities of Cambridge, Birmingham and Sheffield. I am informed that men passing through these laboratories are snapped up by industry with some avidity, and in these days of mourning over so-called over-crowded professions, I would draw the attention both of parents and students to one that cannot find anything like the personnel it demands.

It should be emphasised at the outset that both B. and C. class chemists are of little actual value when they first join the industry, and it is only during their early probationary years when they change from the embryonic pupillary stage to that of worker that one can discover whether they are peculiarly adapted for development work, for laboratory research, or unfortunately misplaced entirely.

What has been said above applies, of course, to the physicist. Indeed, once a scientifically trained man is established in the industry such arbitrary titles as chemist, physicist, or even engineer tend to disappear. The chrysalis, be it chemical or physical in origin, burgeons out into that glorious imago—the petroleum technologist.

From what has gone before it is clear that a careful study of, and good relations with, the various universities is essential. This excellent contact is perhaps particularly evident in the case of the great oil companies who have for many years past taken a keen interest in education generally, and in the development of scientific teaching and thought.

Finally, the preliminary technical training of these recruits is, I am sure, best achieved in a main research laboratory, rather than by posting them direct to the refinery. This is reasonable, because every company has its own special problems relating to petroleum products and often its own methods of testing and refining. It is, therefore, essential that a new man should know something at least about these two major issues before being brought into the refinery. One other aspect which is particularly applicable to large concerns is the social effect; for at the research laboratory, the newcomer will meet many of his future associates, and will be able to gain experience through contact. He will learn that he is a member of a family and not merely an isolated unit. Thus a very real value of the research laboratory lies in its dual function of a training school and a testing ground. There are nowadays

so many fields of work in the industry that it is essential to ensure that a man is on his right road if the best advantage of his services is to be obtained.

Scope of Operations of the Chemical Staff.—After his preliminary training at the research station, there are four main spheres of work :—

1. Fields.
2. Refinery.
3. Development.
4. Research.

Having dealt with the qualifications and types of the recruits, I propose to consider their work under the above four headings in outline, and attempt to bring out the wide and variable nature of their everyday work in these categories.

The Chemist in the Oil Fields.—This chemist operating in the fields has probably the most varied career of all. It should be remembered that his service takes him out into the wilds, with its corresponding attendant problems.

His work will be largely general and routine analysis, but much of this may be other than normal petroleum determinations. For instance, a considerable item in fields analysis is concerned with water, potable for the rigs, and from drilling shows; he has to determine what materials are necessary for the treatment of drinking water and in what quantity. When there is a large staff dependant on the supply, the matter is obviously of the first importance. The fields chemist, further, is a very necessary adjunct to the geologist. Samples of oil, gas and bitumen go to the chemist. He should know something of mineralogy and petrology. He should be familiar with the properties of cement.

In the fields, the chemist is brought in to make many physical determinations for the operator; for example, the oil-gas ratio often has to be determined at a variety of pressures. Gases, either natural gas or that obtained from absorption or compression units, have to be periodically analysed to ensure maximum plant efficiency; whilst another test made on fields gases may be in relation to their physiological effect on operating personnel.

Corrosion occurs in a multiplicity of situations in such an area; in the fields production plant, in casing, and in pipe-lines, in operating plant, leading to trouble in stills and in lines and heat exchangers. The chemist is called upon to determine the extent, nature, cause and cure of the corrosion. Fields plant is always with him, and he will be, as time goes on, more and more concerned with this aspect of his work.

These are some of his major duties. A sideline of great importance is food analysis. Contamination of food is an easy matter in hot climates, and the medical staff need expert analytical assistance; therefore in many fields food analysis is part of his daily routine.

In general, then, a fields chemist needs to be physicist, organicist, inorganicist, chemical engineer, analyst, botanist, metallurgist, bacteriologist; a sufficiently wide range of work to test his resources. Such are the multitudinous duties that the efficient fields chemist performs.

The Refinery Chemist.—The work in the actual refinery is not quite so varied as it is in the fields, but it is sufficiently broad to merit attention. A considerable amount of the chemical work passing through the refinery laboratory is, of course, concerned with the routine work, testing of products, intermediates and crudes. He has to consider, further, the lay-out of the refinery laboratory and so to arrange matters and material that a multiplicity of tests can be carried out with the utmost expedition. Work extends here also beyond the normal routine, for the chemist must be prepared to make analyses of every type of material used in the refinery. Water, water softening, and the multitudinous aspects of corrosion must be considered. The testing of crude oils and products for traces of corrosive substances, the analysis of corroded material, and the effect and use of non-corrosive paints and products, fall almost into the everyday refinery routine. But, indeed, the efficiency of the whole refinery and its equipment is largely his responsibility. For processes such as chemical treatment, desulphurisation, and decolorisation it is necessary for him to lay down definitively the manipulation of the variables time, temperature, pressure and quantity. As for the products obtained, it is his duty to see that they are uniform, that they conform to the standards laid down by his own company, and that they are of sufficiently good quality to merit sales in the competitive markets of the world.

The refinery chemist may be regarded as a buffer State between management, production staff and sales staff, an unenviable position at times.

He is, further, called upon in the rôle of an economist, to invent and develop uses for by-products, such as spent chemicals, acid sludge, coke, and waste gases. Finally, all new plant that is commissioned is under the initial care of the chemical staff.

In our industry, at any rate, executive functions frequently fall to the chemist, and to a young man of sound training and of industrious habit the way of promotion is not too difficult.

We now return to the main research station, the training ground for all the chemical personnel. Here, of all places, should be found

the nursery of new ideas, the source of the most recent information and knowledge, and the first attempts at development of these ideas and this knowledge on to the ultimate operating scale.

The Development Chemist, or the chemical engineer, is in effect the technologist, destined to proceed to the various refineries and to put into practice new and improved methods, operations, processes and plant.

His work is chiefly the extension and furtherance of the work done by the research chemist, to whom I shall shortly refer. The development chemist acts as the intermediary between the research chemist's ideas and full refinery production. For example, a novel method of refining or treatment is originated by the research chemist, who operates it in the laboratory usually in glass apparatus and on a very small scale. The scheme is then passed to the development chemist, who reproduces the small-scale laboratory work on a much larger scale. Model plant is erected and the variables concerned in the process are critically examined. If this test is satisfactory, and it is decided to utilise the new process, then the development chemists will be concerned with the design of plant and ultimately will be posted to the refinery where the process is to be put into operation, and will assist the refinery chemists and refinery staff in the starting up of the new unit. As the new plant comes into production it may be desirable that one or more of the development chemists should stay on at the refinery to see it through its inevitable teething troubles. The chemical engineer is thus a mobile entity, but should always regard the research station as his headquarters. He is seconded to the refinery for plant work.

Clearly, the development staff needs the widest experience in refinery operations to enable them to think in terms of plant. For this reason they must be continually in touch with the refinery staff and share their outlook, and consequently they are bound to exert a steadying influence on research itself. They are a flying squad, and must be available in any eventuality.

These men, in the course of their smaller scale development work, often encounter and solve difficulties which would otherwise only have appeared with probably serious consequences when the full-sized refinery unit was put into operation. The solution of these difficulties in their early stages forms a considerable part of their work.

The close relationship between the development and the research chemist is obvious, and the need for constant contact is clear. The development chemist is the link between the newly conceived idea and the ultimate manufacturing unit.

The Research Chemist.—This group, though relatively small in actual numbers as compared with the other groups of chemists, is

the nerve centre of the staff. To it one looks for the new idea, and the propagation of all that is novel in the field of scientific and technical progress.

The Research Chemists can conveniently be placed into one of two sections,

(1) The Specific or Industrial Research Chemist.

(2) The Fundamental Research Chemist.

In the former category is the man who works closely in conjunction with the development chemists. His work is essentially research in connection with refining and production methods and problems. It is necessary for him to determine the most economical and effective application of mass, temperature, pressure and time in the development of the process.

It is his duty further to study the commercial products of his company and compare them with competitive material to ensure standards being of a sufficiently high nature to give the sales staff every opportunity. An example of this is to be noted in the engine testing of fuels for anti-knocking and other properties. The engines used for these tests, such as the Armstrong, Delco, Thornycroft and Horning, have been devised in the research laboratory. The fuels must be compared with a standard fuel or blend of standard fuels. The problem lies both in the production of satisfactory testing engines and of appropriate standard fuels, which must be constant in properties, and capable of being readily reproduced. Research work in connection with the production of such standard and sub-standard fuel is at present actively engaging the chemist throughout the world. Again, the industrial research chemist is responsible for the invention of new technical products. He is closely in touch with both the production and the distribution sides of the organisation. From time to time he is called in to report on methods and material submitted to the head office staff. These investigations range from the determination of the nature of products both commercial and alleged-to-be commercial.

Oils, petrols, waxes, paints, pigments, varnishes, cements, dopes, etc., come his way, and he is asked to "vet" apparatus including small scale laboratory plant, gas detectors, chlorinators, and in general any material likely to be of service in the organisation.

He acts as consultant to the head office on the value of new patents and processes, and in this connection often has to witness or make tests on new plant and novel operations, and a not unimportant side line is the exposure of those plausible miracle-working gentlemen who haunt the offices of large corporations.

To enable him to cover this wide sphere of activity it is essential that he should be well advised as to publications, patents, etc. It

is, therefore, necessary for him to read widely, and be in touch with all modern technical development.

Lastly, an important company has necessarily to be represented on a large number of committees and societies; this helps to fill up the working day of the industrial research chemist.

We come now to the last group.

The Fundamental Research Chemist.—I rather regard this type as the keystone of the chemical arch, although I would deprecate any attempt to place professional men in some artificial order of merit. His work is basic rather than practical, his task is the elucidation of the unknown. He is an artist rather than a decorator. He is invariably temperamental.

It has just been emphasized that the research man must necessarily keep closely in touch with current thought. This is even more essential for the fundamental chemist, but it is not necessary to emphasize what is natural to him. He must know what is happening in the realms of pure chemical (and physical) research. He must always be on the look out for fresh developments, new syntheses, new compounds, new ideas that may possibly have a commercial bearing, and to these he must draw the attention of his industrial collagues. His industrial colleague on his side must continually drain him of information.

Putting this matter in terms of journals, whilst the industrial research man must read for example the *Journal of Industrial and Engineering Chemistry*, and the *Journal of the Society of Automotive Engineers*, the *Journal of the Society of Chemical Industry*, the *Journal of the Institution of Petroleum Technologists*, his "pure" colleagues must peruse all the Journals of the Chemical Societies, the *Transactions of the Faraday Society*, *The Kolloid Zeitschrift* as types. Similar remarks apply to the physicist who, on the applied side, is an engineer and, on the "pure" side, a fundamentalist.

It will be obvious from the above that the research station must be near a great city that possesses universities, scientific and technical societies, libraries and, in general, culture. A body of scientific men working in juxtaposition will tend to suffer from mental inbreeding. I have indicated that in the first place fluidity of staff should be maintained, and the station should be, if not a spiritual, yet a mental headquarters for the whole of the scientific personnel of the company. In the second place every advantage should be taken of the cultural facilities afforded. The staff should be encouraged to read papers, to attend meetings, and to mix with their fellows. As a counsel of perfection there might be urged the desirability of withdrawing a man from the industry and returning him to his university for a refresher course. This Institution has

played and is playing an important part in this direction, and it would be difficult to over estimate the stimulus that it gives to members who are fortunate enough to be able to be present at its deliberations.

We have now traced the opportunities that lie before an apprentice chemist. I would say, in conclusion, that these opportunities are vast, and that their lot is fortunate. We live to-day in the era of petroleum, and the possibilities of the extension and development of this era are immense. The chemist is in the fore-front of this expansion, he is the spear head of the attack. As one who has worked many years in the industry, who has watched the careers of many of its young recruits with interest and sympathy and appreciation, and who frequently has counted himself specially fortunate to have been born in this oil age, I wish all my colleagues far flung in the fields, refineries and laboratories in every continent the good luck they deserve.

Vivant, Crescant, Floreant.

Sir Frederick W. Black said that the President's address was not open for any discussion at the time of delivery, but later, when they read it in print, as he was sure all would with attention, they could form their own considered opinions. He did not think that the President need make any apology for speaking of the chemical side. It was the very essence of their existence as an Institution that they recognised at least three main branches of science and technical training. There had been discussion in that room as to what proportion the engineer contributed to the main results and they had had chemists opinions also. It had been extremely interesting to hear Dr. Dunstan's description of the specialised organisation of the chemist's work in relation to every branch of the oil industry. He supposed it was in the memory of many present that not so many years ago people talked about science as something to be pursued for its own sake, and hardly at all as to what practical value it might have for the good of mankind. He could remember listening to an address some years ago by that great man whose death they had been deploring —Lord Balfour—who emphasised the point of pursuing science for science' sake. In after years it fell to Lord Balfour to give a great initiative to the movement for industrial scientific research. Lord Balfour's name, he thought, would long be associated with many developments of that kind.

In years before the war it was said that some of the Continental nations had developed the industrial application of science to a

greater extent than had this nation. To-day, however, we stood abreast of any nation in the world that was applying science to industry. (Applause.)

He proposed a hearty vote of thanks to Dr. Dunstan for his presidential address. He congratulated him also on his re-election as President a second time. (Applause.) Those who served on the Council in particular, and he thought he might say the members of the Institution at large, knew what a vivid and vigorous and enthusiastic personality they had in Dr. Dunstan. He had brought all those qualities to bear on the work of the past year, and he would bring in the second year the same qualities in at least an equal degree to the work of the Institution to its very great advantage. (Applause.)

The vote of thanks was carried with acclamation.

The President briefly replied, and the meeting closed.